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MATERIALS EVALUATION FOR SERVICEABILITY OF OPTICAL GLASSES UNDER PROLONGED SPACE CONDITIONS

June 1967

FINAL REPORT

Contract NAS9-4845

Avco Document TR 67-G-109-F

AVCQ ELECTRONHCS DIVISION TULSA OPERATION TULSA, OKLAHOMA

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FOR EWORD

This report was prepared by the Avco Electronics Division, Tulsa Operation as the final documentation of work performed under Contract NAS9-4845 issued by the National Aeronautics and Space Administration, Manned Spacecraft Center. This program was monitored by Dr. W. R. Downs of the Manned Spacecraft Center. This report covers work performed from July 1965 through June 1967.

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N O T I C E

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SUMMARY

Samples of prospective Apollo window materials were exposed to temperature cycles of $+250^{\circ}$ F to -250° F and solar simulation approximating the Johnson spectrum. Two ninety-=daytests were conducted, The purpose of the test was to determine the optical degradation resulting from exposure with periodic measurements of the spectral transmittance, Considerable degradation was found to occur in the plastics and polymers with no observable degradation occurring in the glasses. Spectral transmittance curves of each material, made in situ, are presented for the two ninety-day tests.



I. INTRODUCTION

This contract was issued to determine the serviceability of the window systems for the command module and the lunar excursion module for extended missions in space. The specific objective was extending information concerning infrared and visible light: transmissibility of the window materials to the mission times expected. Two ninety-day tests were to be conducted, Window materials supplied by NASA for the tests are presented in Table I and Table 11.

Sample Holder No.	TABLE I Test No. 1 Material
1	Owens Illinois Inorganic Polymer #650
2	Dynasil Pure Fused Quartz 1/411
3	Owens Illinois Inorganic Polymer #100
4	Dynasil Pure Fused Quartz 1/8"
5	Corning Photochromic Glass
6	Corning 7940 (coated) Outer Window 1/16"
7	LEM Chemcor Uncoated Code 0312
10	Acrylic Sheet 1/411
11	New Chemcor Uncoated Code 0313
12	Corning Special IR Glass (anhydrous composition)
13	Copolymer Styrene
14	Corning 1723 (coated) Inner Pane 1/4 ¹¹ C/M (alumino-silicate)
16	Cross-linked Methacrylate, 1/411 thick
17	Corning 7940 (uncoated) 1/2" (fused quartz)



TABLE II Test No. 2

Sample Holder No.	Material	
110.		
1	Owens Illinois Inorganic Polymer No. 650	
2	Dynasil Pure Fused Quartz 1/8"	
4	Libby Owens (Coated)	
5	Corning Photochromic	
7	LEM Chemcor Uncoated 0312	
8	Corning 7940 (coated) Outer Window 11/16 ¹¹	
10	Acrylic Sheet 1/4"	
11	New Chemcor Uncoated 0313	
13	Copolymer Styrene	
14	Corning 1723 (Coated Inner Pane 1/411)	
16	Cross-linked Methacrylate	
17	Corning 7940 (Uncoated) 1/211	
19	Libby Owens (Uncoated)	
20	Owens Illinois Inorganic Polymer No. 100	

This work was initiated to expose the samples to the ultraviolet portion of the spectrum principally, and measure damage in the visible and infrared portion of the spectrum. This was to be measured on various glasses, plastics and polymers. The test required the simultaneous exposure of the glasses to a vacuum environment while being temperature cycled with LN₂ for one hour and heated for one hour. The samples were mounted in a specially designed sample holder capable of holding up to 20



samples. The samples were mounted in a circular ring which could be rotated by means of an external lever and the solar beam diverged to a size which covered all the samples in the ring. A pressure of 10⁻⁸ torr was attained in the chambers when no samples were installed; but the plastic samples outgassed and drove the pressure up to 10⁻⁷ torr.

The transmittance of the samples were measured periodically in-situ by ports at either end of the chamber. These allowed the standard lamp beam to pass through the samples and into the monochromator. Spectral energy distribution of the source was measured at each sample position prior to beginning the test and a photocell monitored the output of the solar simulator during the test. Details of the test equipment and techniques are discussed later.



II. NATURE OF OPTICAL DEGRADATION MECHANISM FOR WINDOW MATERIALS **EXPOSED** TO TEMPERATURE AND SOLAR SIMULATION

Little information has been found on experiments conducted under temperature and solar ultraviolet irradiation, especially on glasses. Experiments conducted on solar irradiation of polymers (1) reported that films of commercial phenyl silicone, vinyl chloride and methyl methacrylate polymers underwent appreciable cross-linking on exposure equivalent to a few days in space sunlight. At exposures corresponding to a week or two in space, other polymers were discolored, in some tests, and lost most of their mechanical strength and elongation of flexibility.

Measurements on a polyester, for example, indicate that after the first few hours in which little change occurred, 2000 to 3000 Å radiation five times more intense than in space, increased the absorption coefficient at 4300 Å about 2 cm⁻¹ for each factor of 10 increase in time of exposure. A relation of the form mentioned implies that most of the darkening would occur in the first few weeks of exposure to sunlight.

Certain other tests were conducted with ultraviolet irradiation of plastics to measure changes produced in the tensile strength and elongation (2).

Most glasses will undergo color center development and corresponding loss of optical transmission on exposure to sunlight that will be encountered in space⁽³⁾. Glasses which show a decrease in light transmission after exposure to the radiation of a mercury vapor lamp actually regain some of their transmission after exposure to longer wavelength radiations^(4,5).



III. TEST REQUIREMENTS AND CONDITIONS

The requirements of this contract were that satisfactory curves of visible and infrared light transmissibility be obtained for each specimen of glass composition under the combined environmental conditions. It was required that transmission curves be obtained

at the commencement of test work; after 14 days' exposure from start date; after 30 days' exposure from start date; after 60 days' exposure from start date; and after 90 days' exposure from start date.

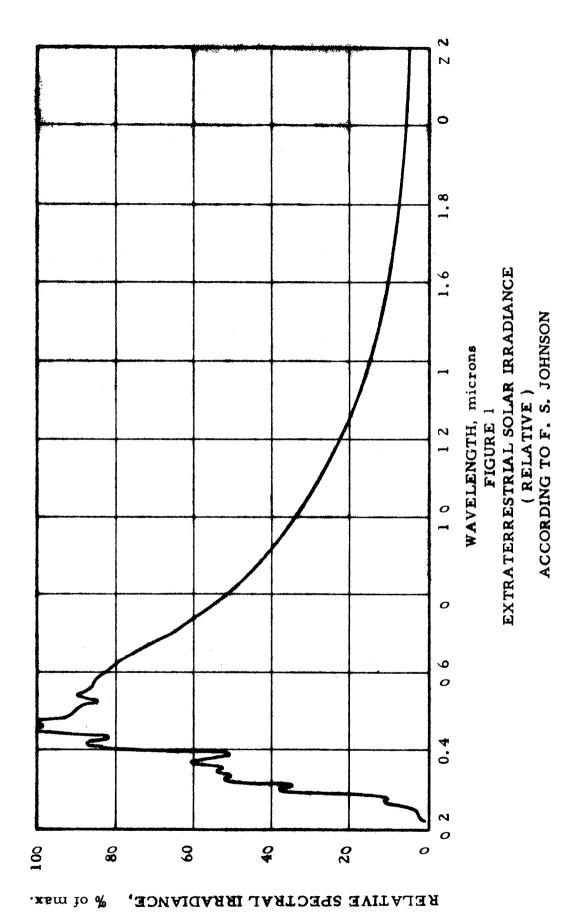
The sample glasses were furnished by NASA and were cut to size by the Contractor. The samples were to be temperature cycled under exposure condition of

-250°F to +250°F Pressure to 10⁻⁸ torr Time: 90 days continuous

The samples were to be continuously irradiated with ultraviolet irradiation extending down to 1650 angstroms, and vacuum conditions for earth and lunar orbital conditions. The extraterrestrial solar irradiance (Relative) according to F. S. Johnson was used as a goal. A plot of this spectrum is shown in Figure 1. According to this spectrum, the peak extraterrestrial spectral irradiance occurs at about 4500 angstroms, dropping to insignificant values at about 2000 angstrom and 4.0 microns at the short and long wave ends of the spectrum, The xenon arc lamp with a quartz envelope radiates strongly down to 2500 Å with a Suprasil envelope (Englehard Industries product) the range can be extended somewhat since these high quality ultraviolet grade quartzes pass radiation to approximately 1650 Å.

In addition to the normal testing a cold soak test was performed on the first test only. The test was performed in conjunction with the 60-day transmittance test. The samples were allowed to come to near room temperature and the 60-day transmittance measurements were made. These measurements were used as a baseline for the cold soak to follow. The temperature controller was then turned to the coldest extreme and the LN₂ allowed to flow.







This was allowed to remain in this condition for twenty-four hours. At the end of the twenty-four hours, the transmittance measurements were made. At the end of this time the coldest temperature measured on the copper cups was $-266^{\circ}F$ and the warmest was $-199^{\circ}F$,



IV. DESCRIPTION OF EQUIPMENT AND TECHNIQUES

The test setup is as shown schematically in Figure 2, and Figures 3, 4, 5, 6, and 7 are actual photographs of the setup. The system consists of an (1)ion pumped vacuum chamber in which is mounted a rotatable sample holder disc bearing against a heating and cooling plate; (2) a solar simulator in which is mounted a 5-KW xenon lamp; (3) a lamp power supply; (4) a test equipment console; and (5) a monochromator and standard lamp for making transmission measurements of the sample in line with the entrance and exit port of the vacuum chamber. To monitor the pressure in the vacuum chamber, a Varian Model No. 971-0014 pressure controller is connected to a Bayard Alpert Ionization Gauge Tube. The heating and cooling plate is controlled by temperature controller designed and fabricated at Avco which senses the plate temperature and activates a solenoid to electrically heat the plate during the one-hour cycle or connect the cooling coils to a large reservoir of LN2 during the cooling cycle. The electrical output of the monochromator is indicated on a Hewlett Packard Model 400D voltmeter. Supporting power supplies are also mounted in the rack to provide the photomultiplier tube high voltage and standard lamp voltage.

The standard lamp used was a General Electric bulb rated at 6 volts and 18 amps.

The solar simulator consists of a vertically mounted 5KW xenon lamp with a collector mirror at a chosen distance from the lamp. The collector mirror is a 13-1/2 inch diameter ellipsoidal reflector manufactured by Bausch & Lomb, Inc. and is intended for use in theatre movie projectors. It came with a reflective coating on the second surface which was not suitable for this application since the glass would absorb the ultraviolet radiation, needed to simulate the Johnson spectrum. This was remedied by having the first surface aluminized and overcoated with silicon monoxide. The geometry of the reflector is such that when used with the image and object distances chosen for the systems, the light from the xenon arc source comes to a focus in the form of a ring covering the circular array of samples. This was desirable since less radiant energy was wasted in the area not occupied by the samples. The simulator housing was cooled with blowers and adjusted to maintain proper temperature on the lamp.

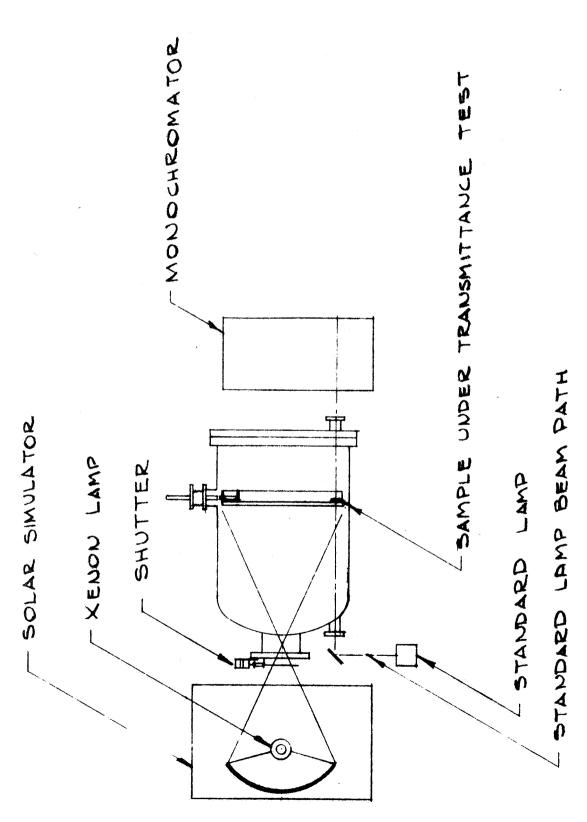


Figure 2

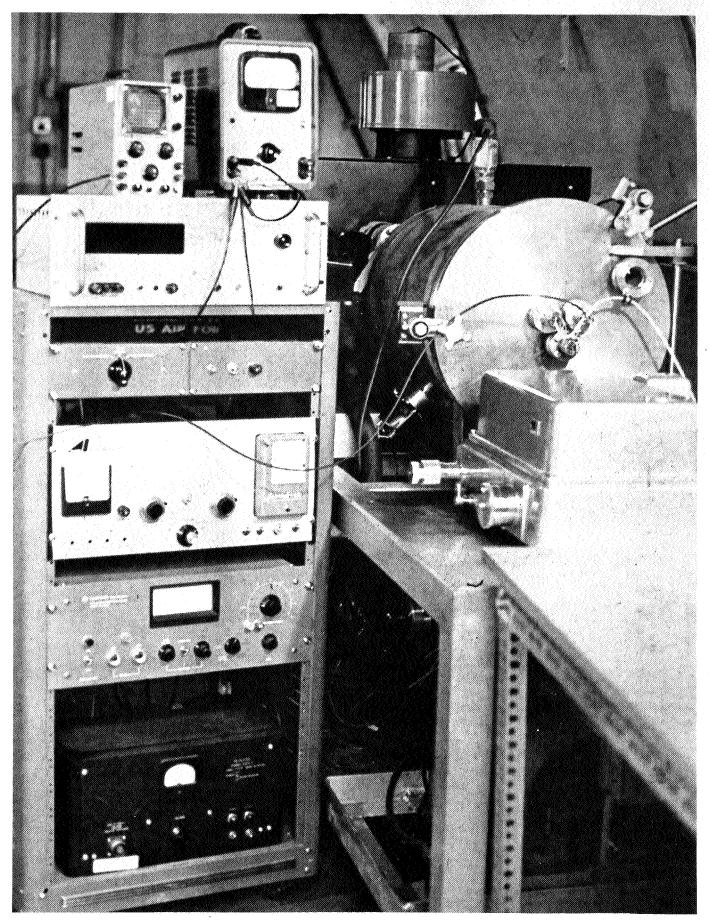


Figure 3 -- Test System

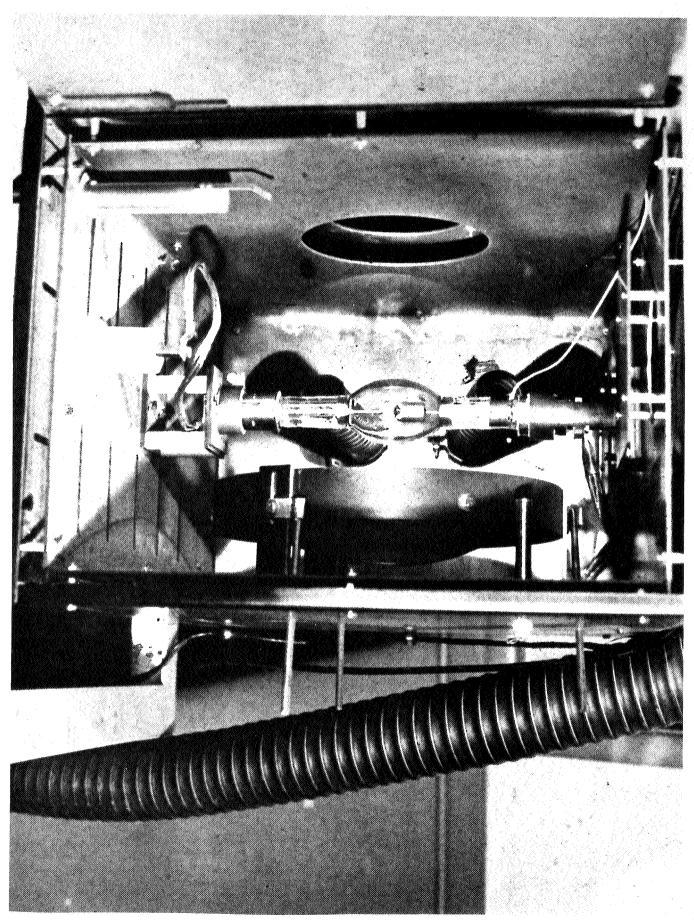
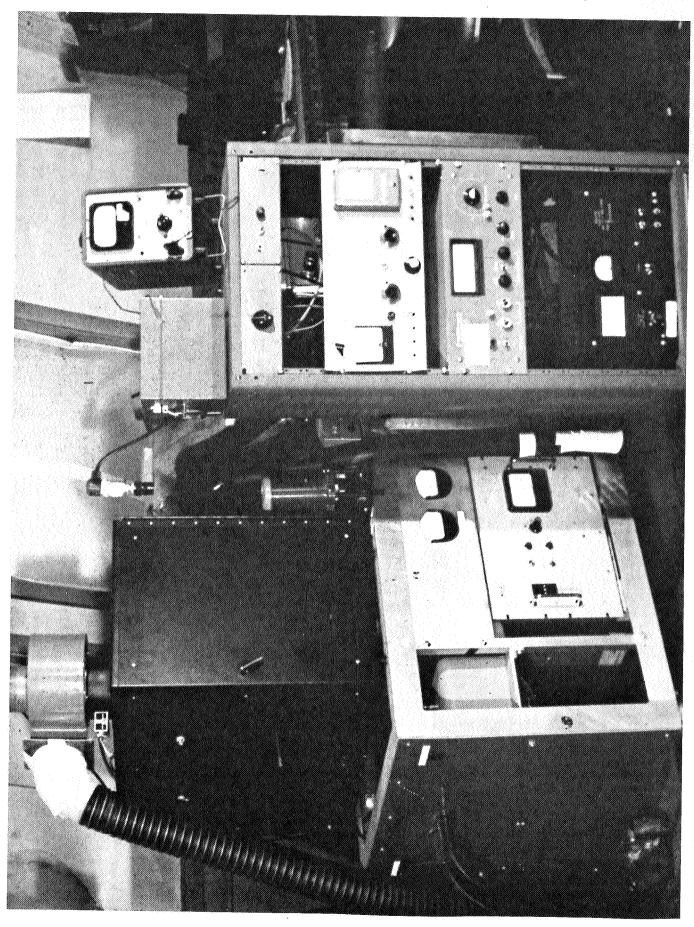


Figure 4 -- Xenon Lamp- Solar Simulator



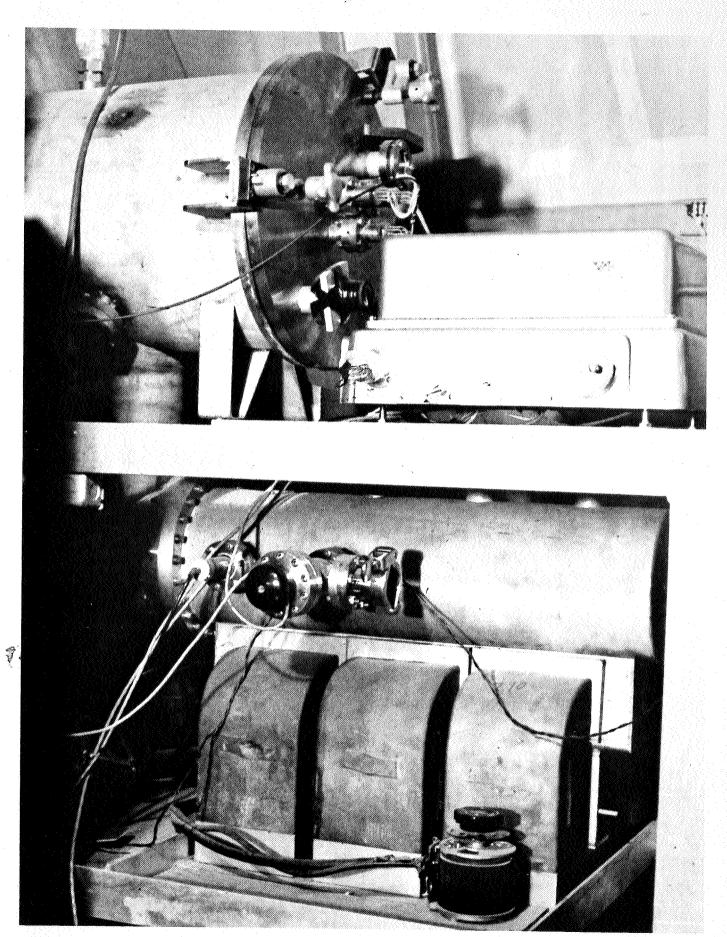


Figure 6 -- Ion Pump - Test System

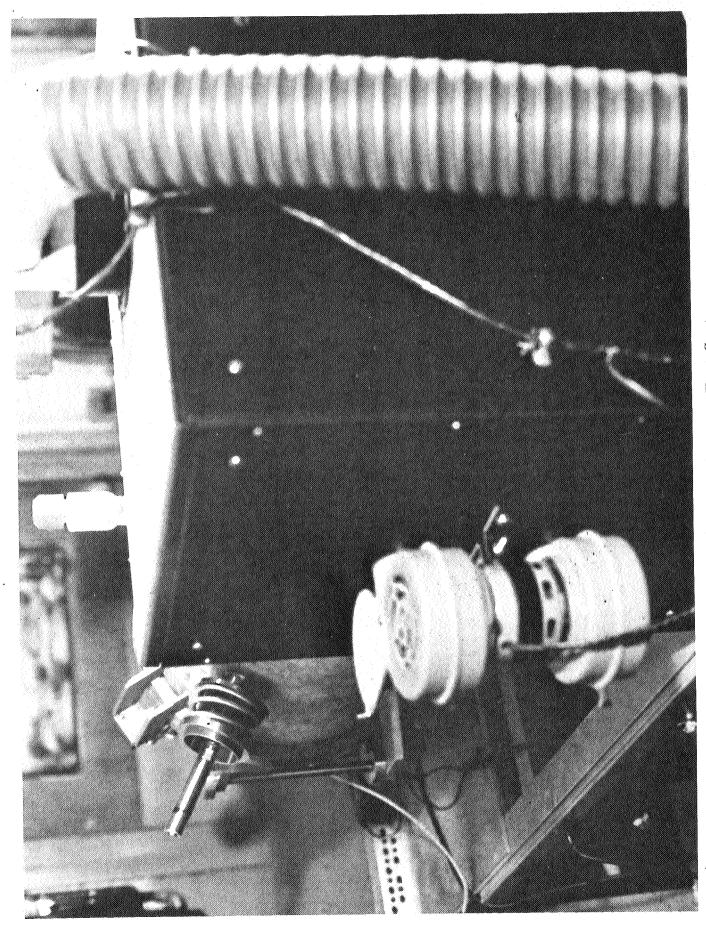


Figure 7 -- Advancing Lever - Test System



During the cold cycle a shutter which was actuated by the same signal as the LN₂ solenoid valve, dropped in front of the entrance window on the vacuum chamber. This was to block the solar energy during the cold cycle.

The temperature of the samples were monitored using a L & N Model 8651 potentiometer connected to thermocouples mounted in the sample holder cups. The sample holder cup surfaces which bear on the large copper heating and cooling plate and its surface were both hand polished to provide maximum bidirectional transfer of heat, To prevent galling of the copper-to-copper surfaces during rotation of the samples, the surfaces were coated with a vacuum compatible lubricant, Tungsten Diselenide. The inner surfaces of the cups were also hand polished to provide maximum transfer of heat.

The transmittance measurements were made using a Perkin-Elmer Model 99 chopped single beam, double pass prism monochromator. The detectors were an RCA 1P28 photomultiplier tube, for .4 microns to .7 microns, and a Perkin-Elmer 012-0352 lead sulphide cell detector, .7 micron to 2.5 micron, These detectors were mounted in the monochromator and resistance heating was employed to maintain the monochromator housing at constant temperature. A focusing lens was added to the output port for the standard lamp on the vacuum chamber in order to focus the parallel beam of light from the standard lamp. This caused the beam to diverge and adequately cover the detectors so that positioning of the prism or detector was not critical.

All test equipment bears current calibration stickers from a local calibration service traceable to the National Bureau of Standards.

Initial Calibrations

Before mounting the sample holder ring and cooling plate, spectral distribution measurements were made at each sample position. The requirement was to approximate one sun of total integrated incident energy. The calibration measurements were made with a Hy Cal pyrheliometer mounted on a test fixture so it could exactly duplicate the location of the individual samples. It was found that the beam



was not very uniform in intensity and the samples were rotated each day so that they received as nearly as possible equal amounts of integrated energy. The measurements around the periphery of the chamber in the sample holder locations are tabulated in Table 111.

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TABLE III

Sample **	Relative
Position	<u>Intensity</u>
1	. 6
2	1.1
3	1.1
4	1.1
5	1.3
6	1.2
7	1.2
8	. 9
9	.6
10	. 2
11	. 2
12	. 6
13	, 8
14	1.0
15	1.3
16	1.2
17	1.2
18	1.2
19	1.2
20	1.1
21	. 2

*Relative to 1 Sun

[&]quot;"The center of Sample Position, 1 is approximately 17° clockwise from the 360° position looking in the end of the chamber.



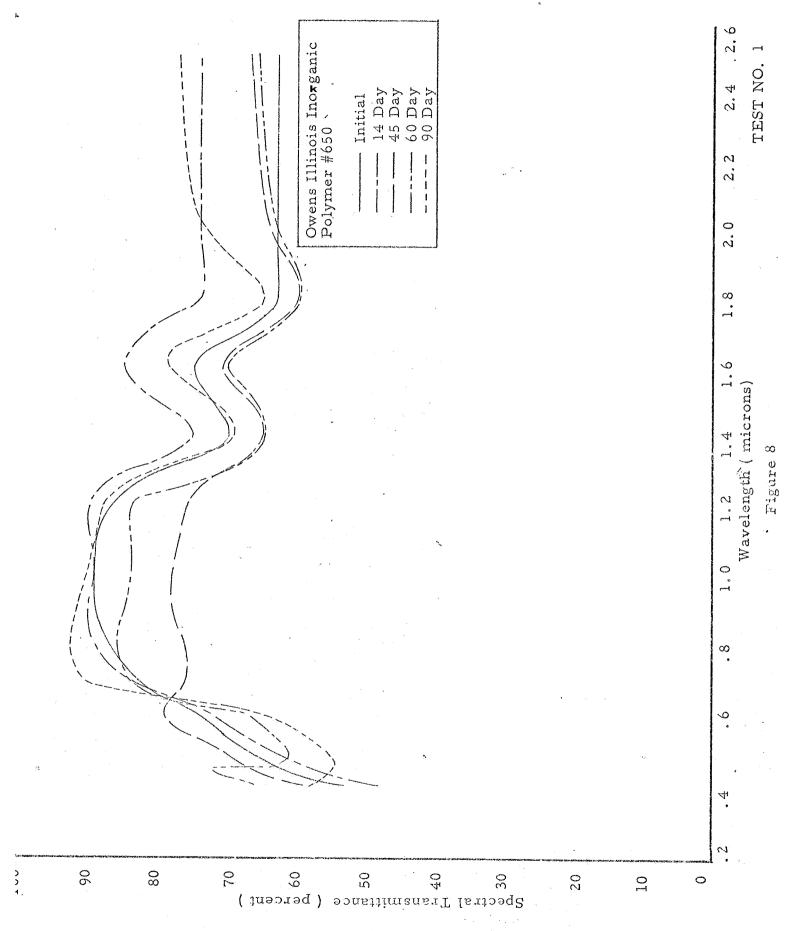
The sample holder, samples, cooling and heating plate were installed and the vacuum chamber was pumped down to the 10⁻⁸ torr region. The temperature controller was connected and the cycling time of one hour for each half the cycle was allowed to occur. At the conclusion of each 1/2 cycle, the thermocouples were readout and recorded. The hottest cup attained a temperature of +259°F and varied down to +220°F. While on the cold cycle, the coldest temperature attained was -221°F and varied down to -179°F.

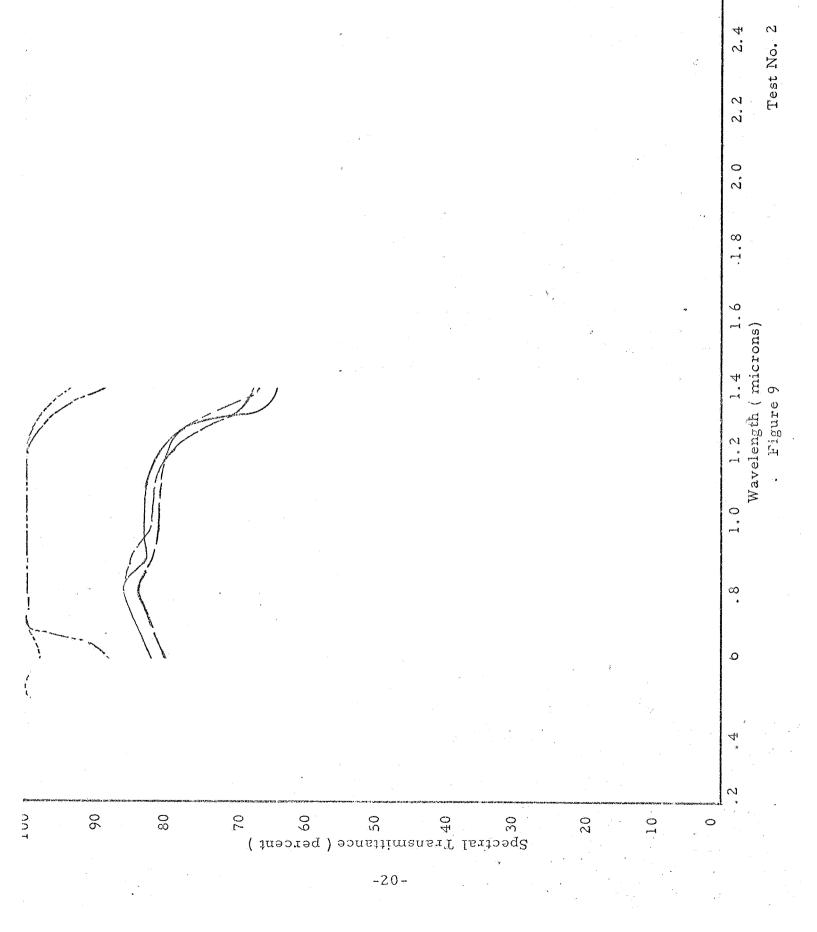


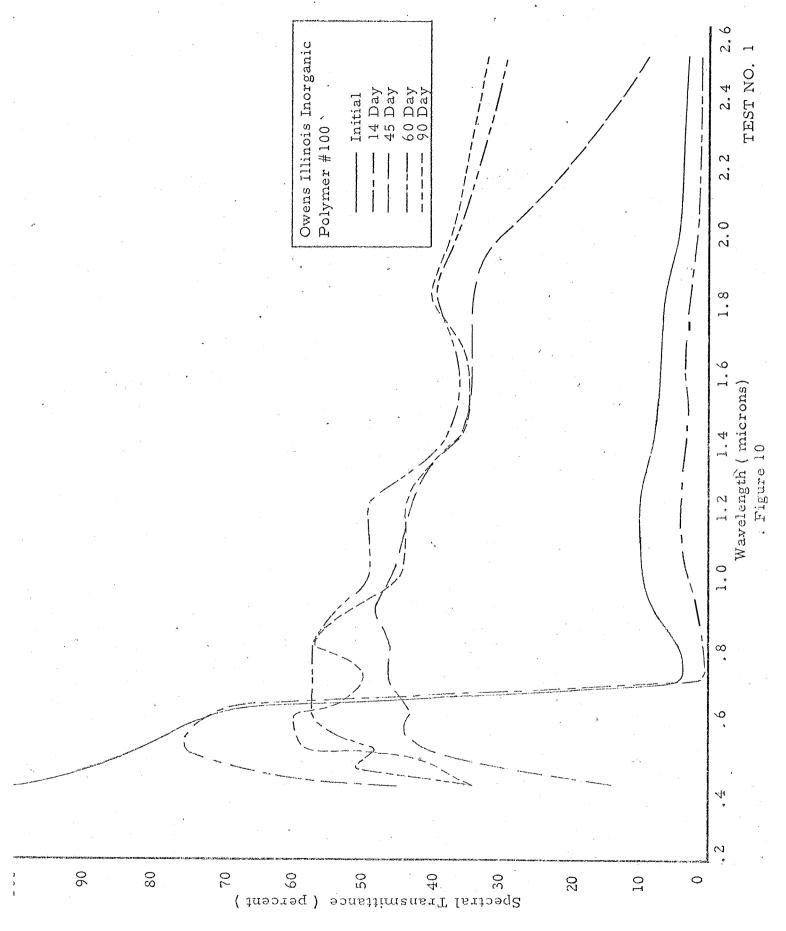
V. RESULTS

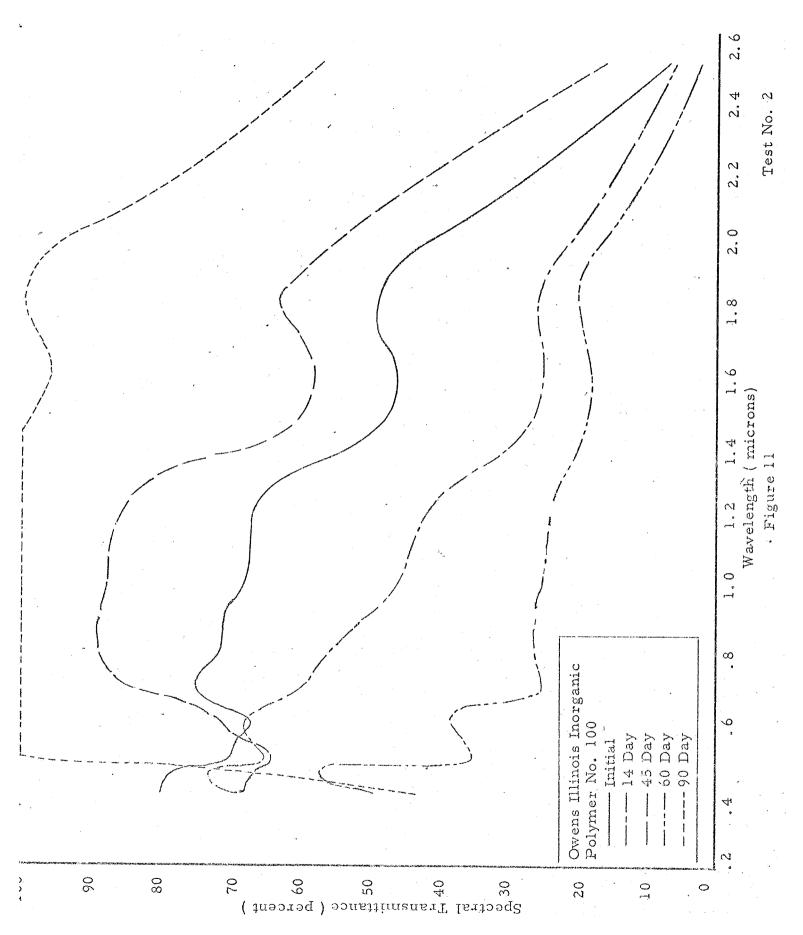
The transmittance curves for the materials tested in the first and second 90-day test are given in Figures 8 through 35. These results were obtained by:

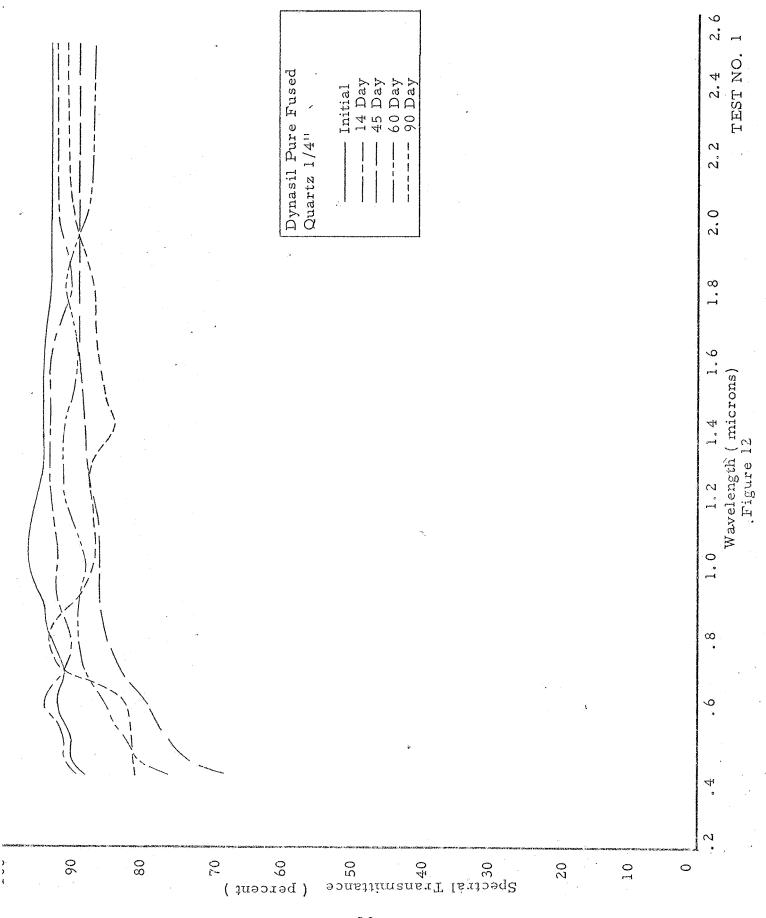
- 1. The sample holder was rotated until a blank hole was lined up with entrance and exit potts of the vacuum chamber.
- 2. The standard lamp measurement was made over the spectrum required. The peak of the photomultiplier output was recorded at the beginning and end of the readings to determine if the photomultiplier shifted during the measurements.
- 3. The sample to be measured was rotated, with the advancing lever, into line with the standard lamp entrance and exit port of the vacuum chamber. A window at the opposite side of the chamber permitted visual observation of the material under test.
- 4. Two materials were recorded and then another standard lamp measurement was made through a blank hole. This standard lamp reading was compared with the initial standard lamp measurement. If there was any difference beyond the stated accuracy of the system, the measurement was repeated.
- 5. If the system was under test the cycle was suspended and the samples allowed to come to room temperature.
- 6. In the case of the cold soak, the measurements were made while the samples were at their cold temperature and the cycle was in the cold position.

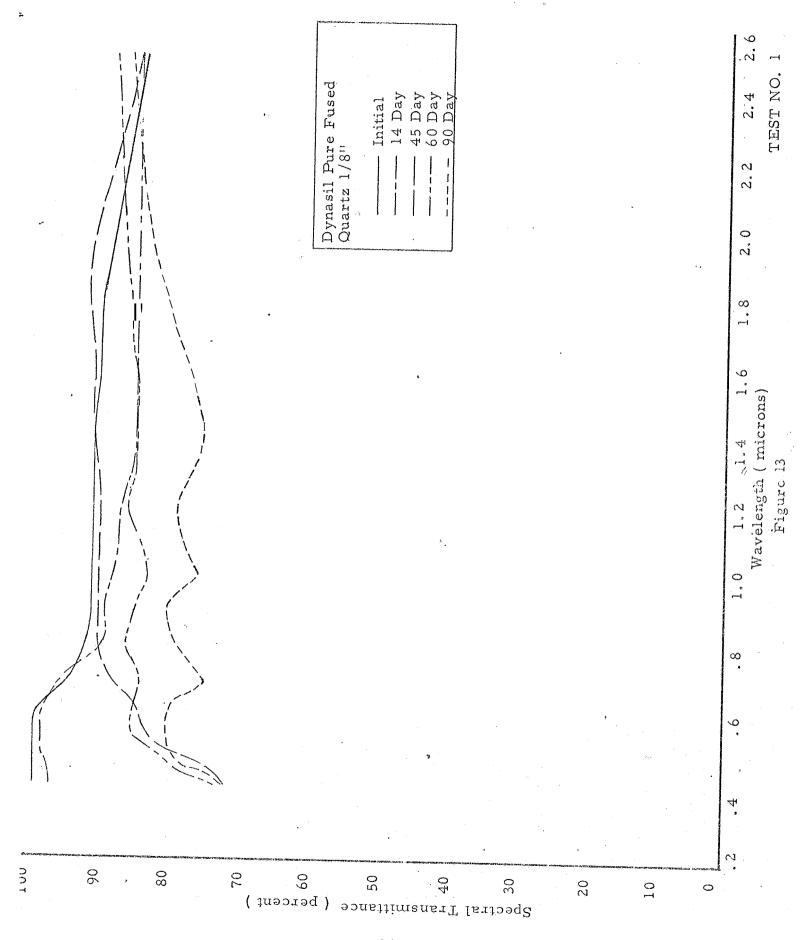


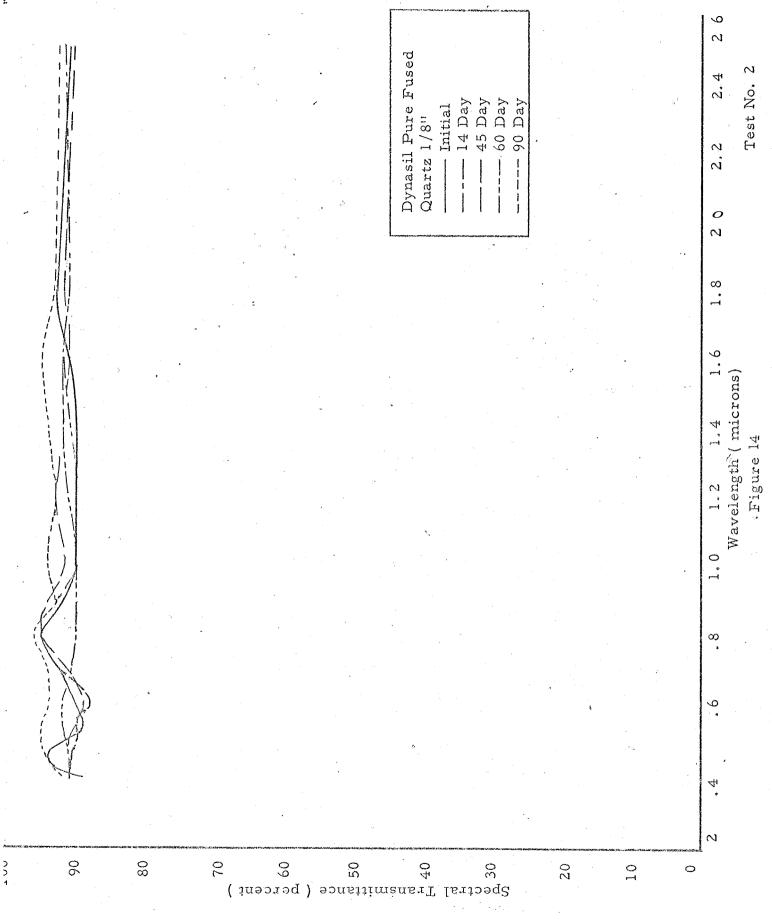


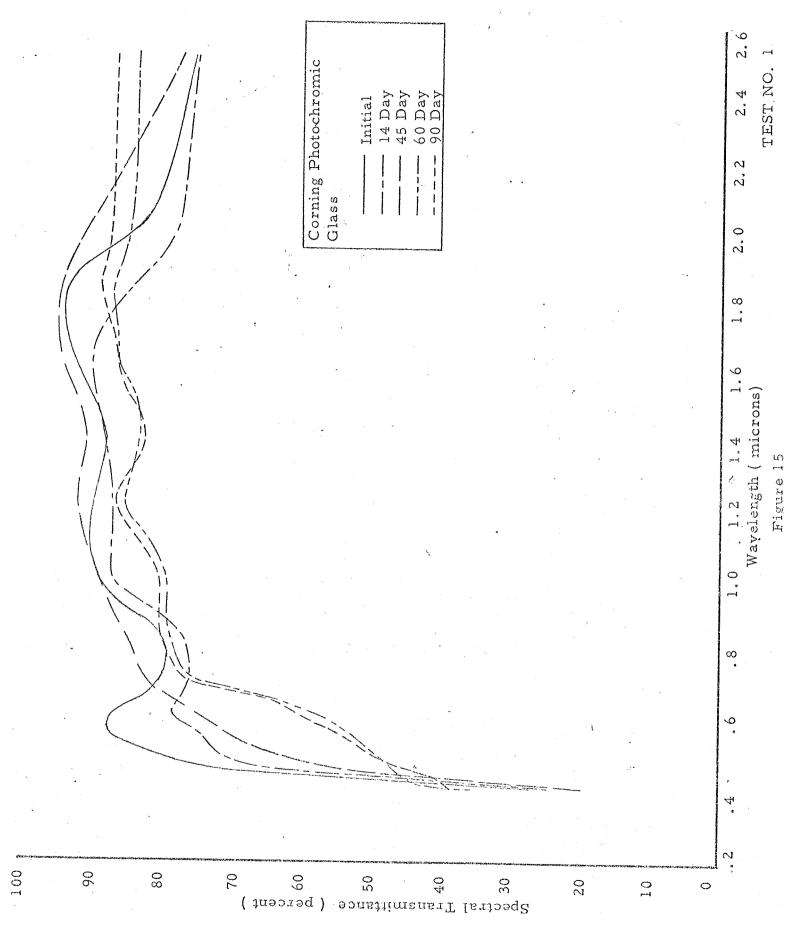


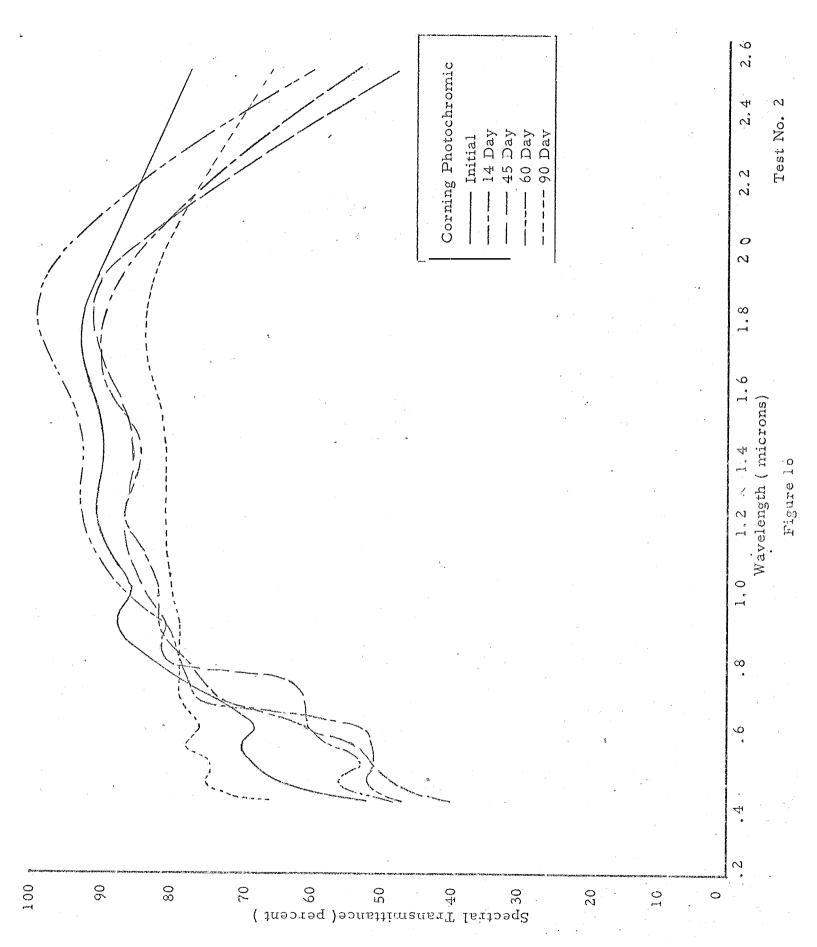


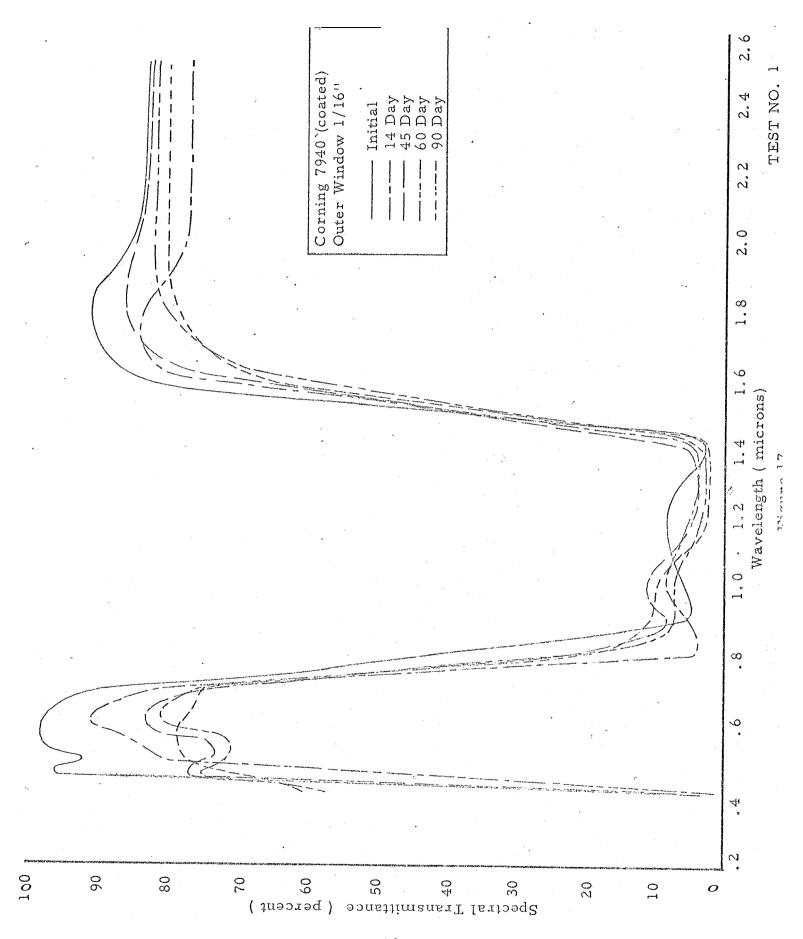


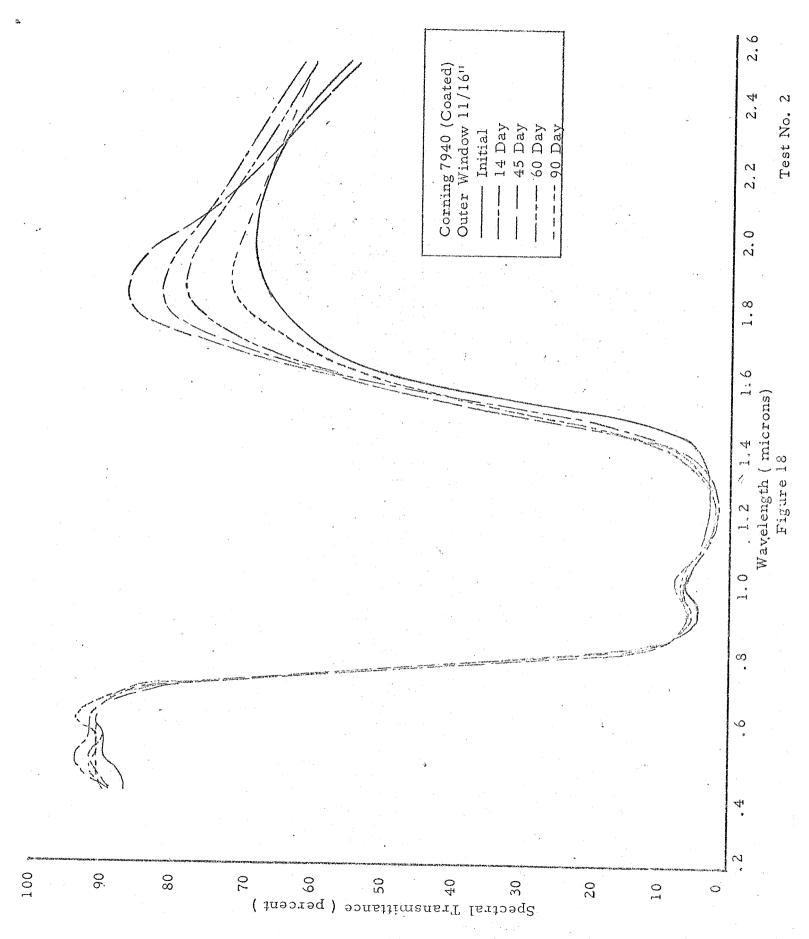


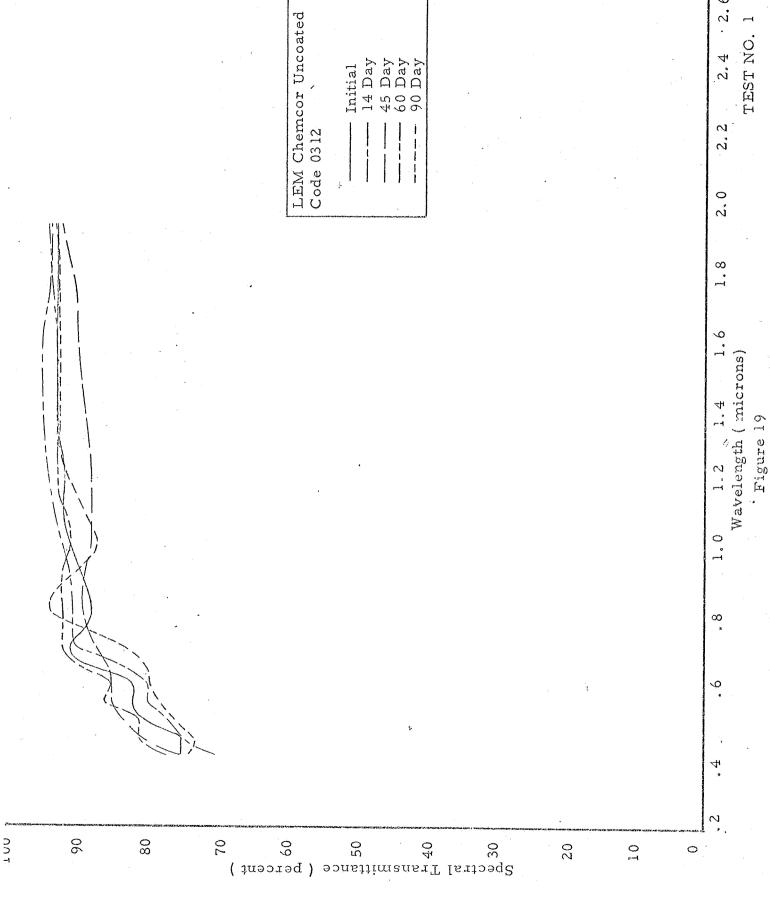


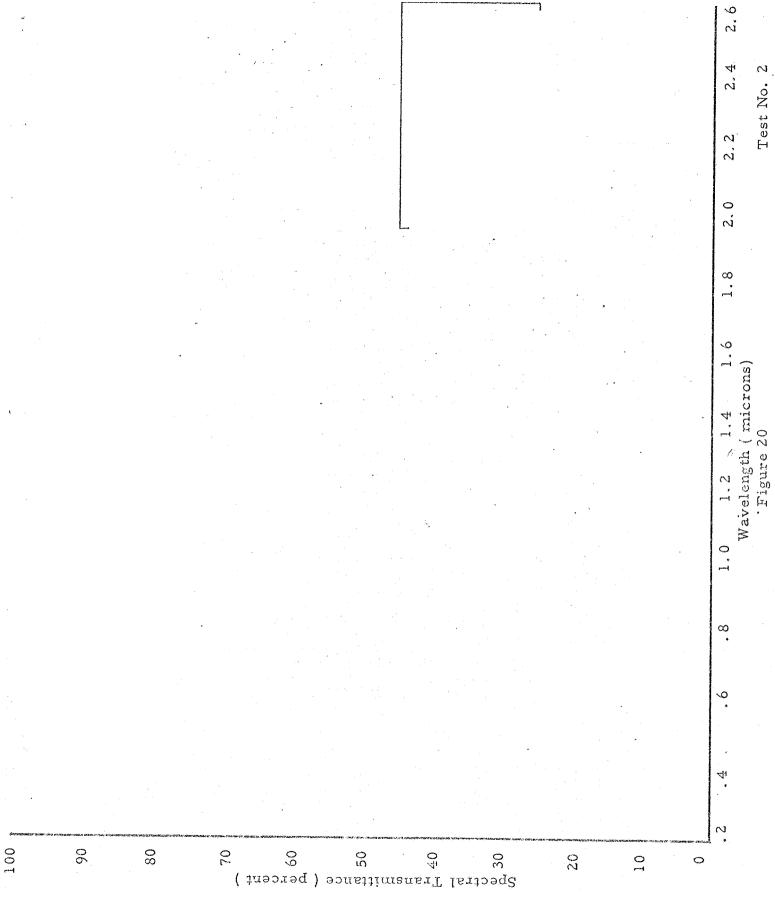


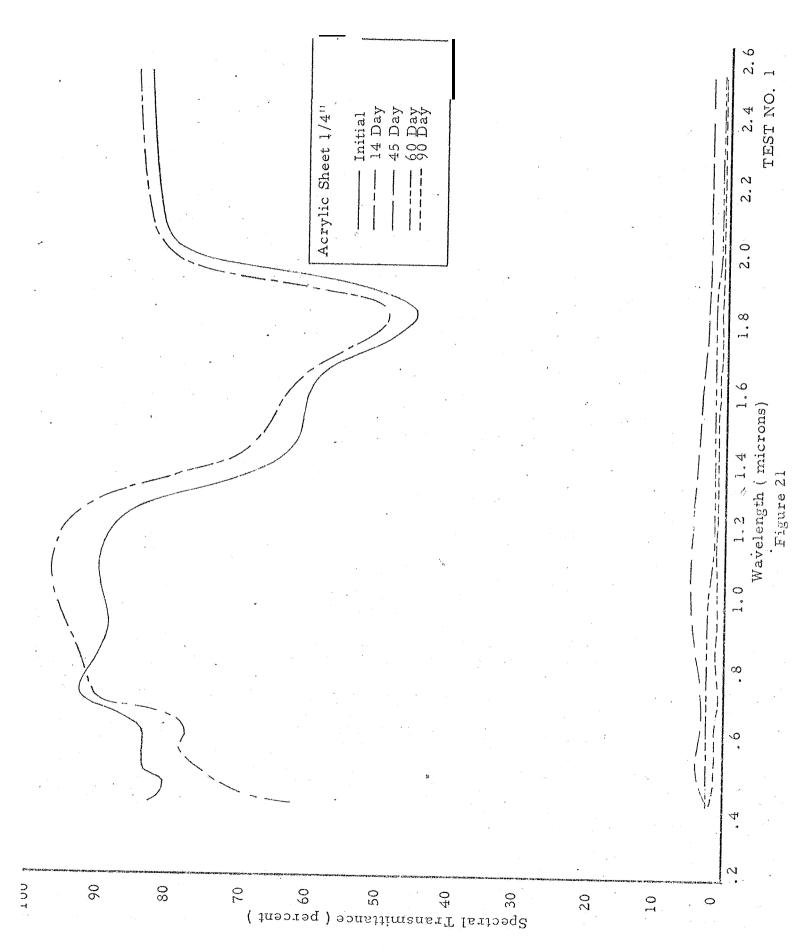


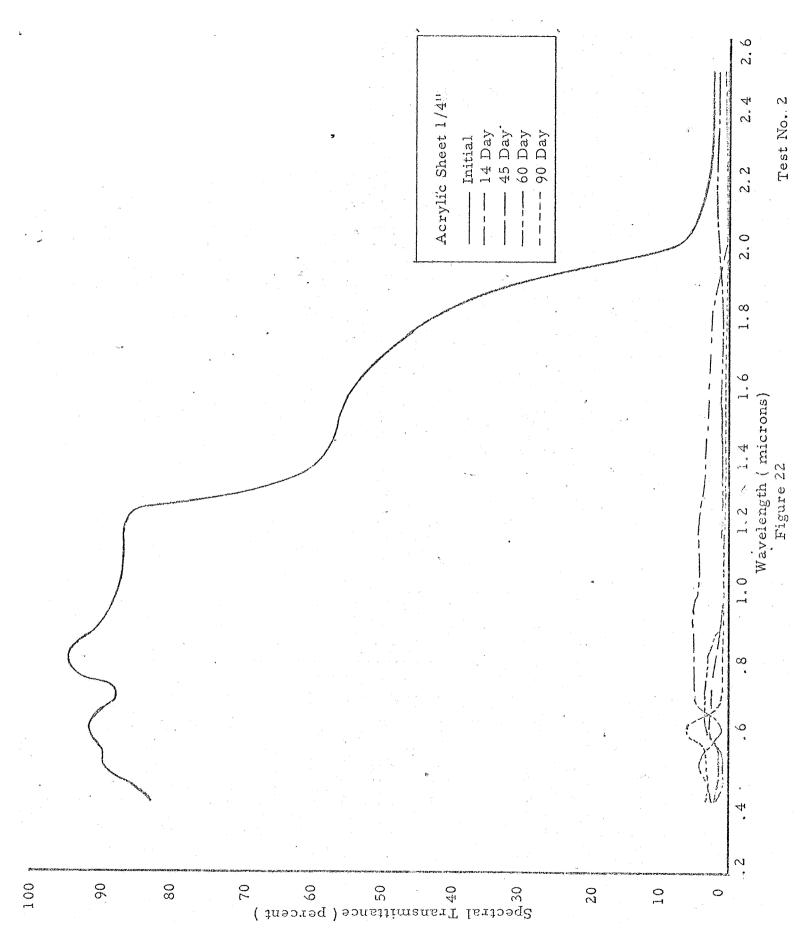


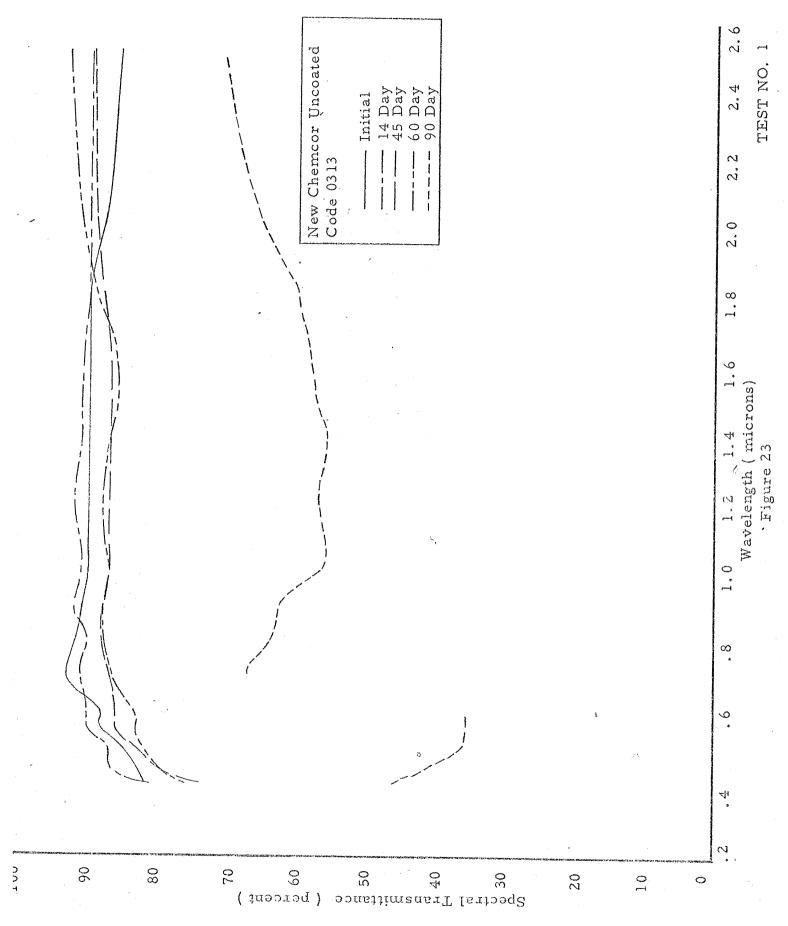


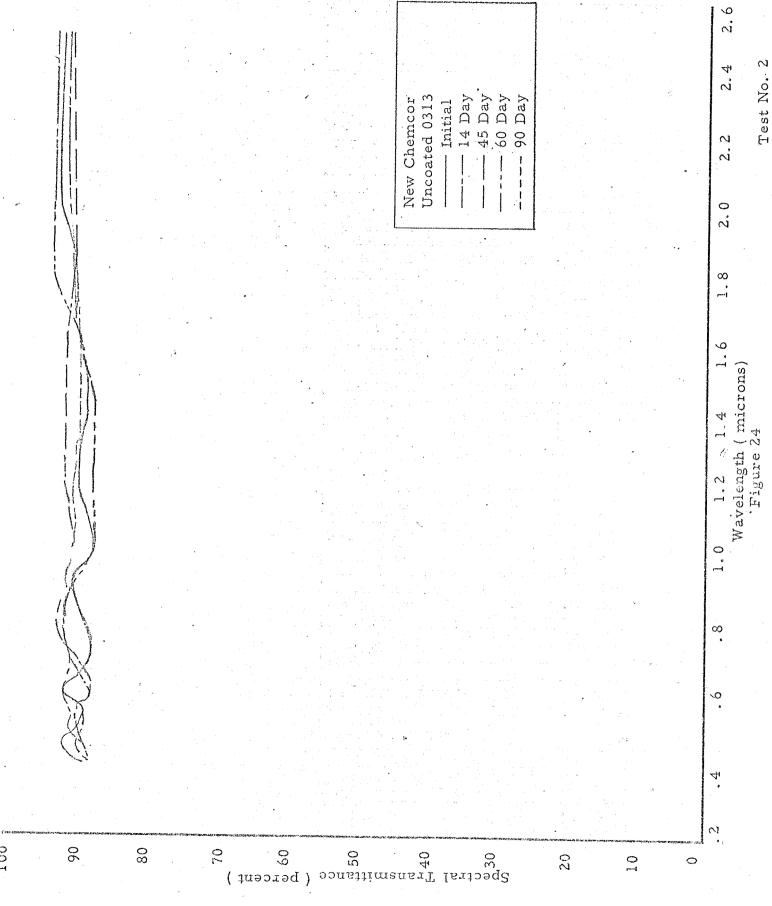


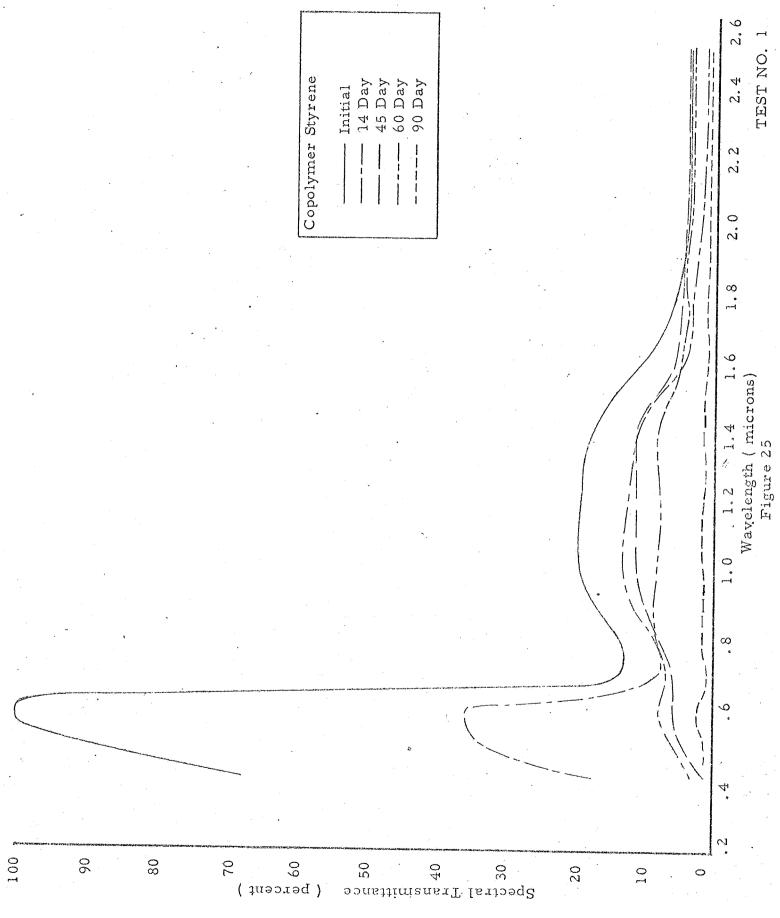


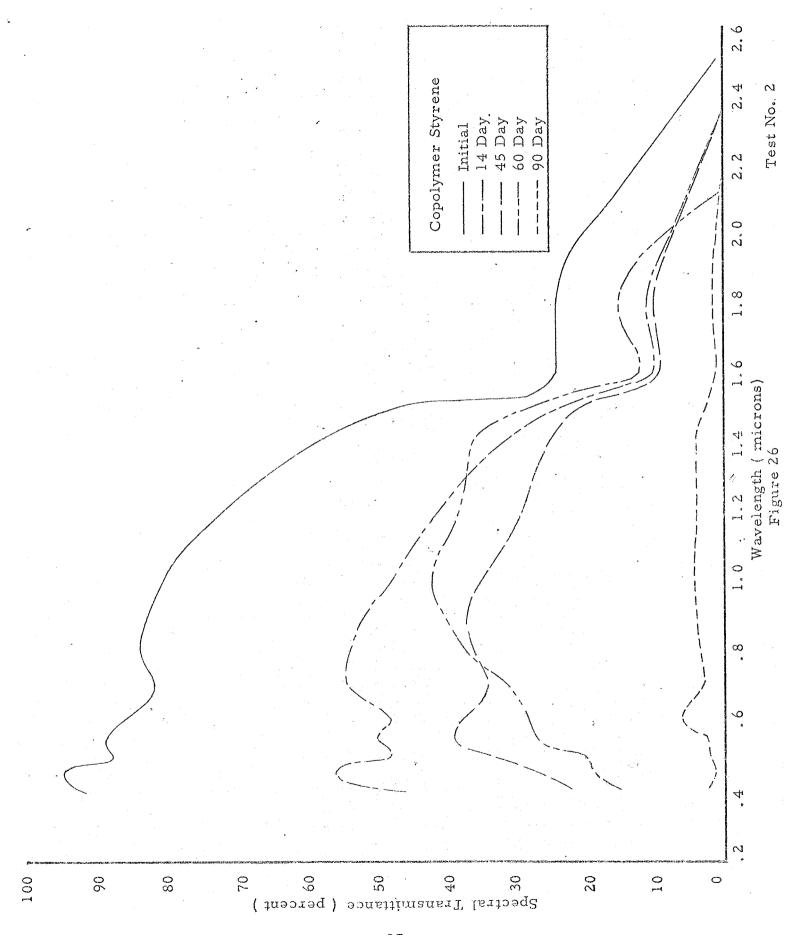


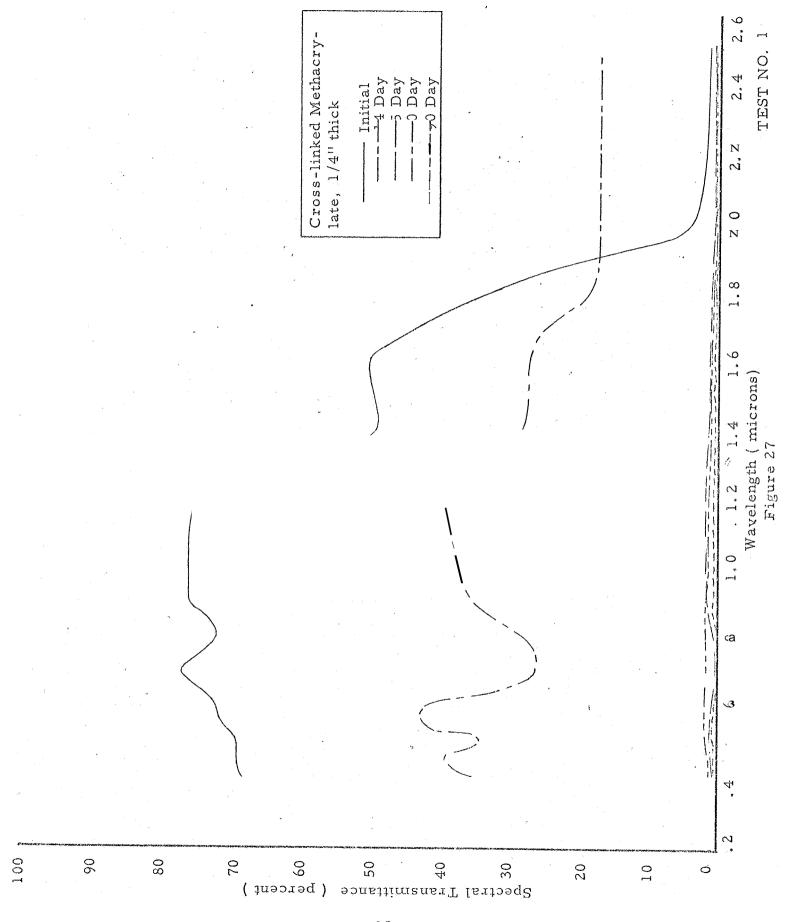


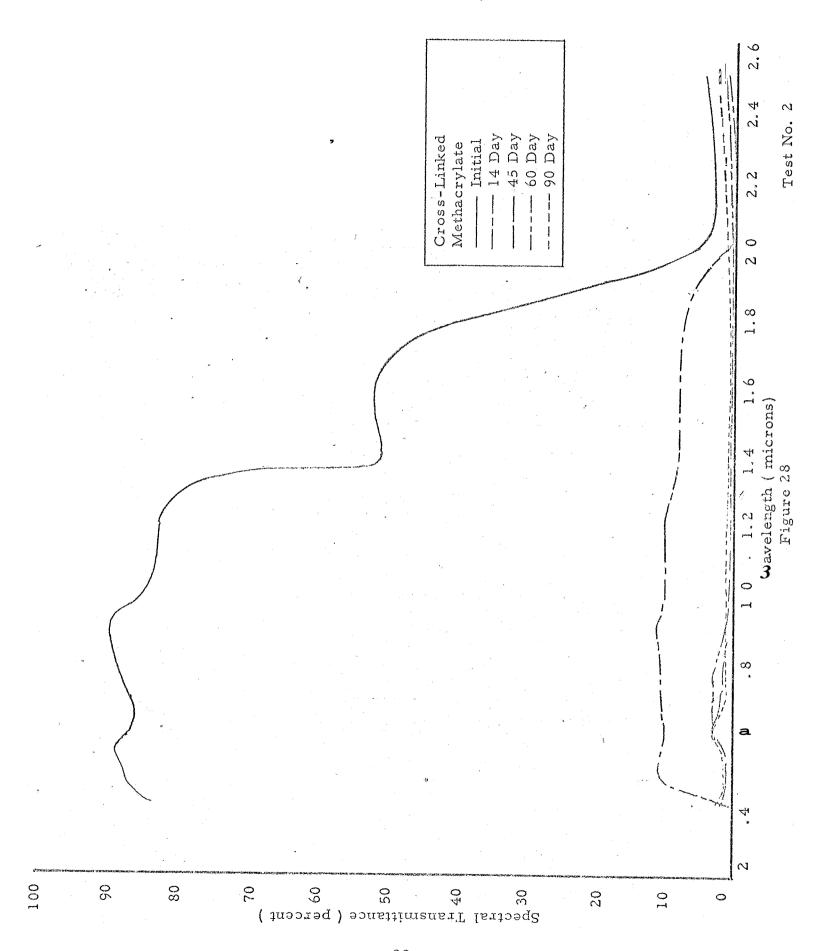




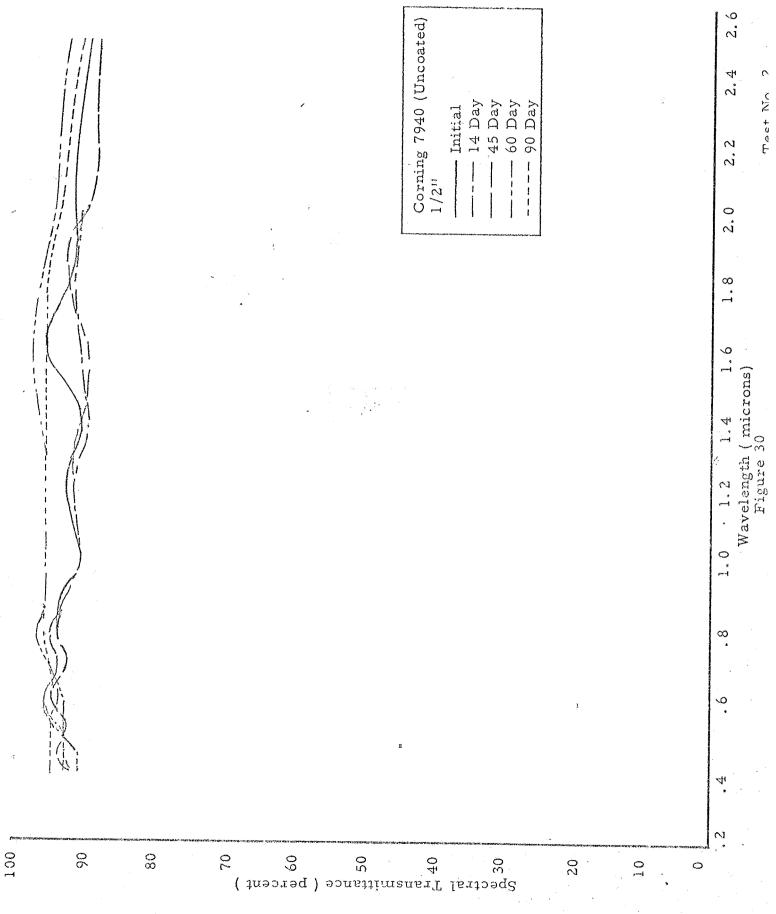


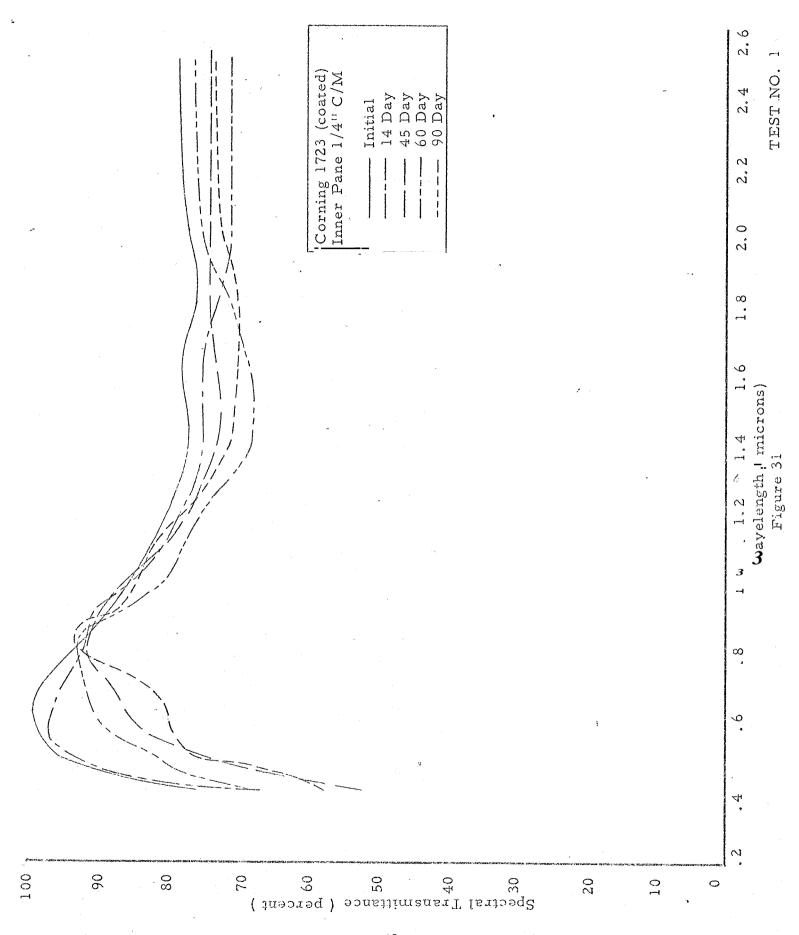


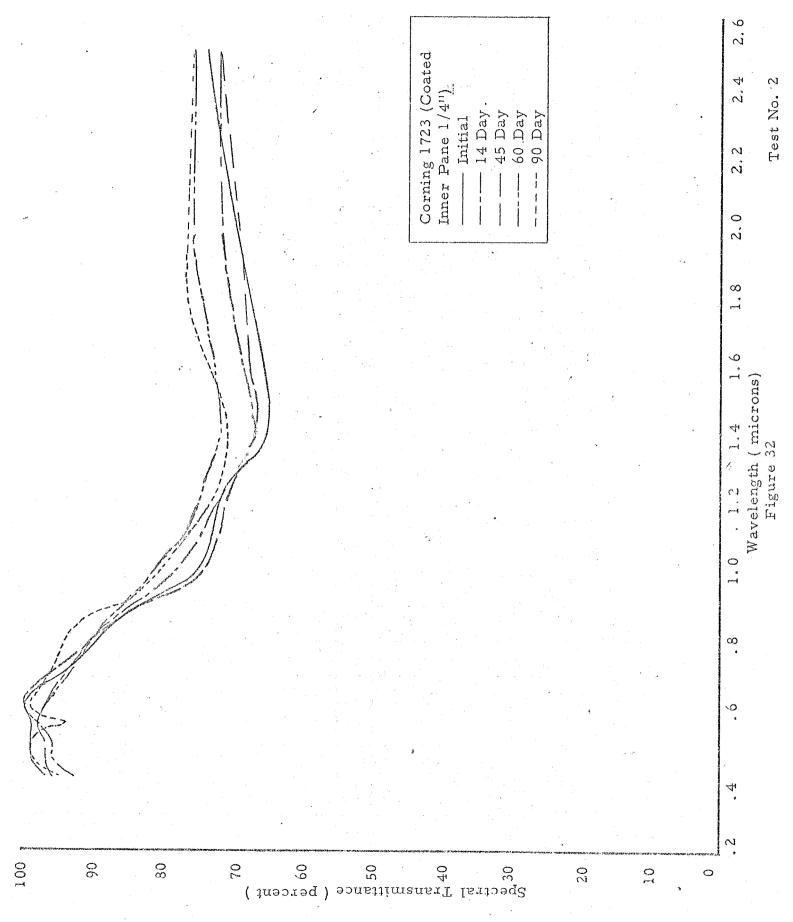


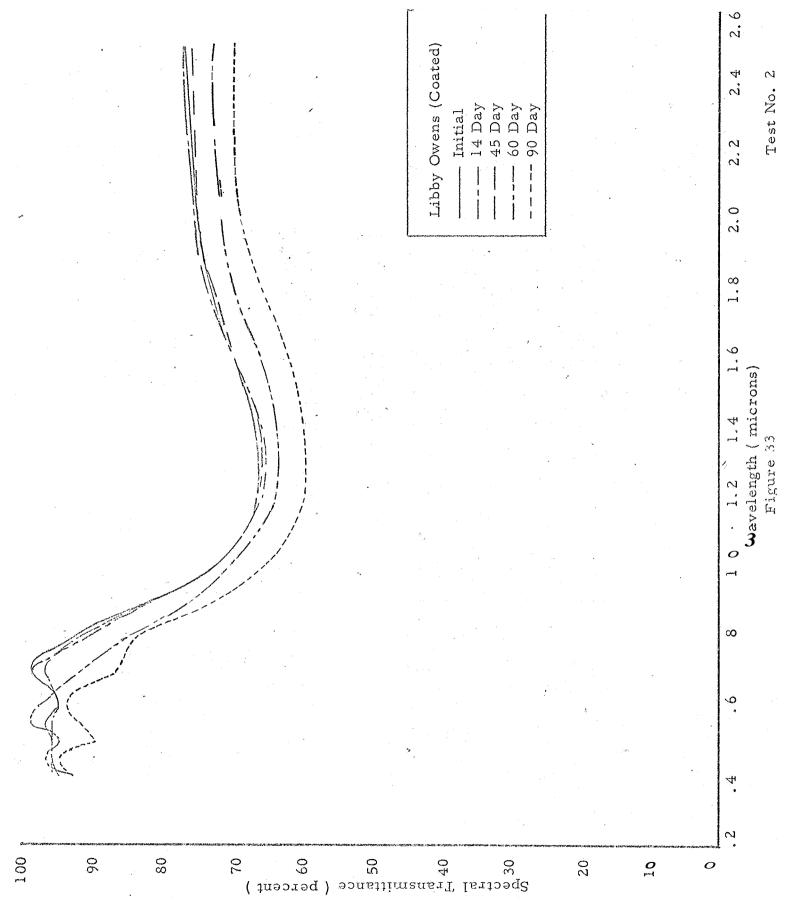


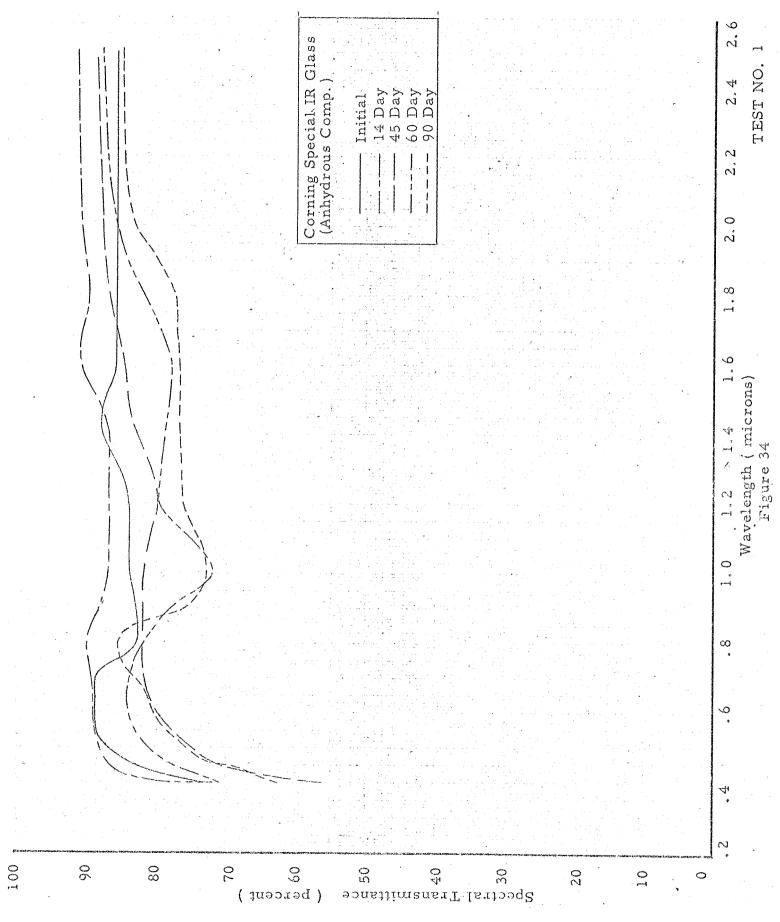
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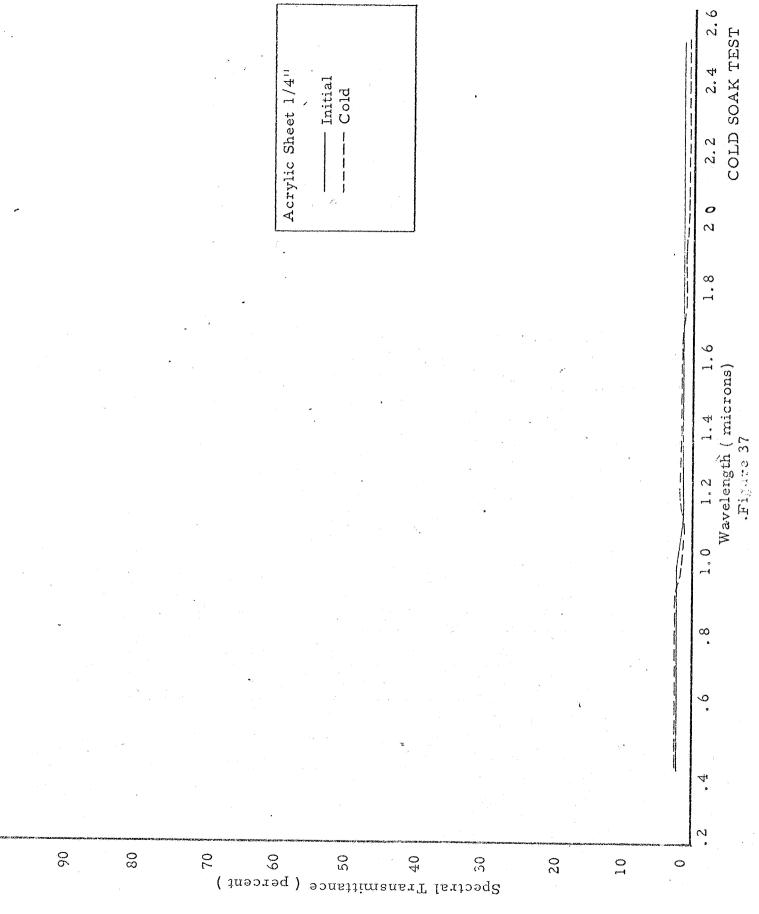
In addition to the data shown here, a Plexiglass 55 sample was attempted but the first temperature excursion to $+150^{\circ}$ F caused such deformation and outgassing that the sample had to be removed from the chamber and returned to the Technical Monitor.

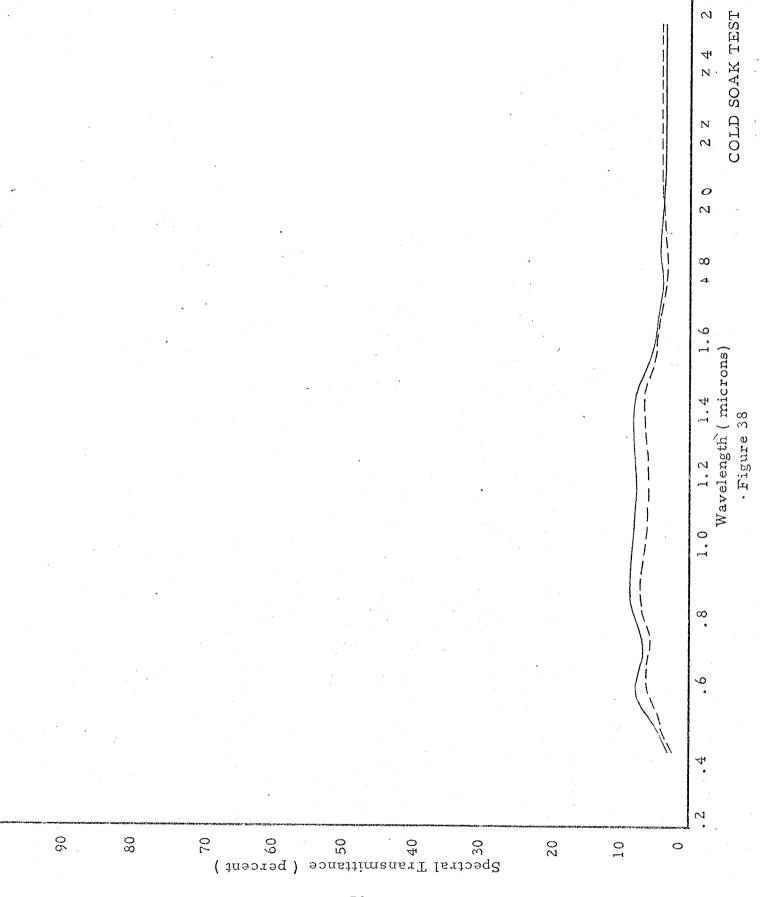
The results of the cold soak are shown in Figures 36 through 49 The results were fairly consistent in showing no effect on transmittance due to cold temperatures,

The results of both tests, were fairly consistent so that the second test served to confirm the results of the first test.. The second test was more conclusive because it is felt the vacuum chamber was cleaner after a SO-day run.

The equipment accuracy is assessed at .+3%. This is due principally to the detectors used in the monochromator. The repeatability of the detector from one exposure of light to another varied in short periods of time but when the light was left on the detector it was stable to better than one percent. Three detectors were procured and all had similar results. Where questionable results were obtained from the detectors, the tests were repeated or a standard lamp measurement was made very close to the measurement of the sample. This established a relationship for the stability of the detectors. The photomultiplier repeatability was similar to the Lead Sulphide cell.

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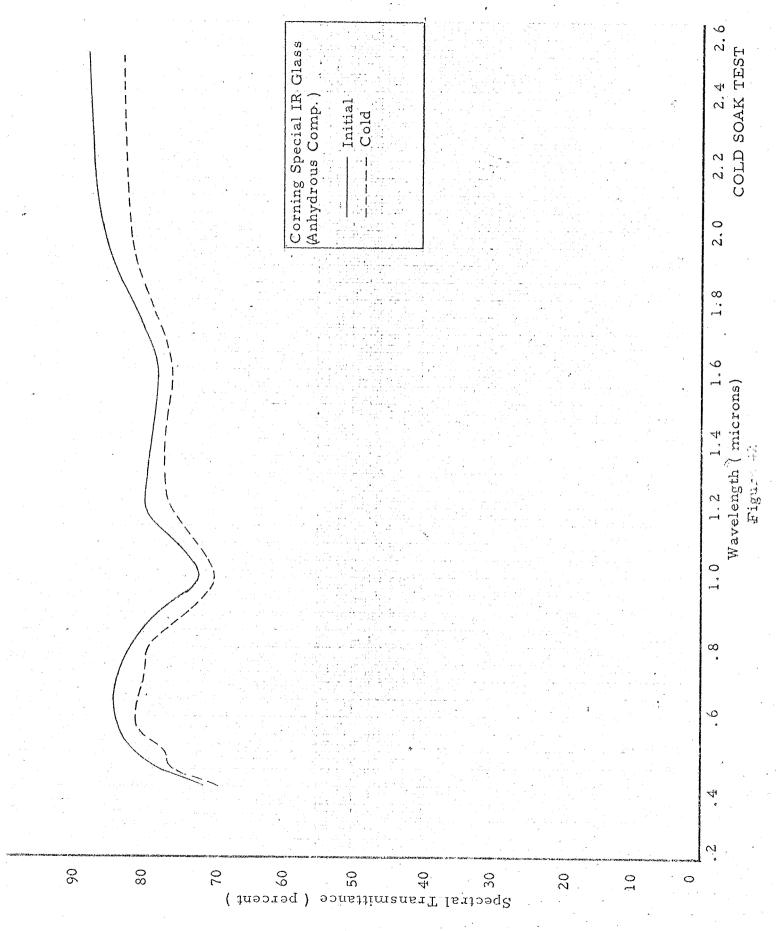
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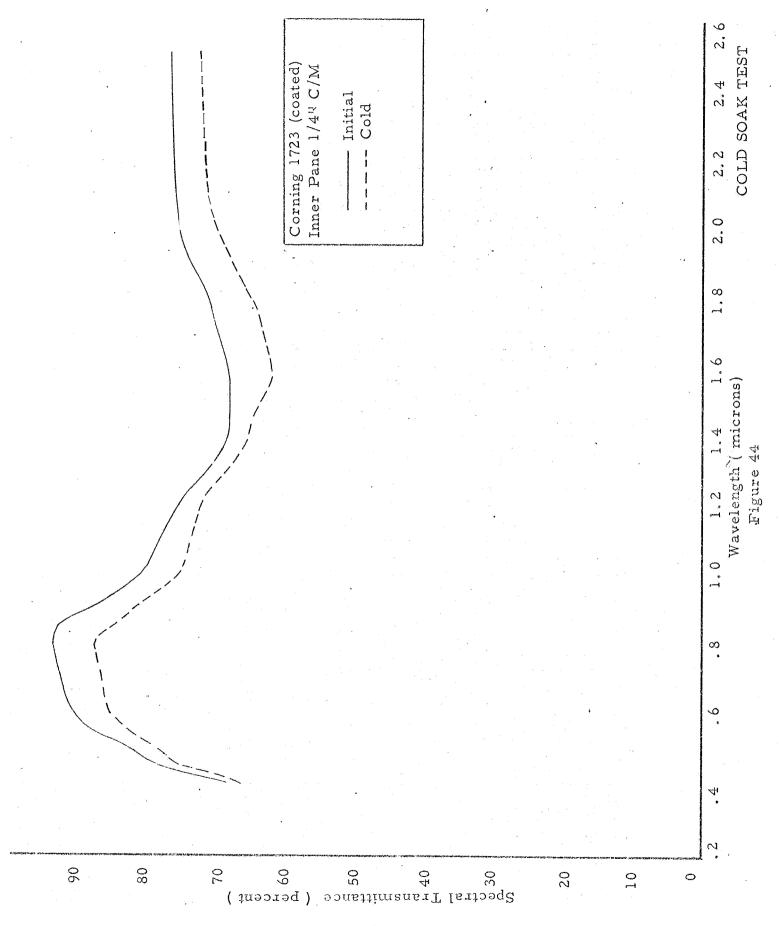
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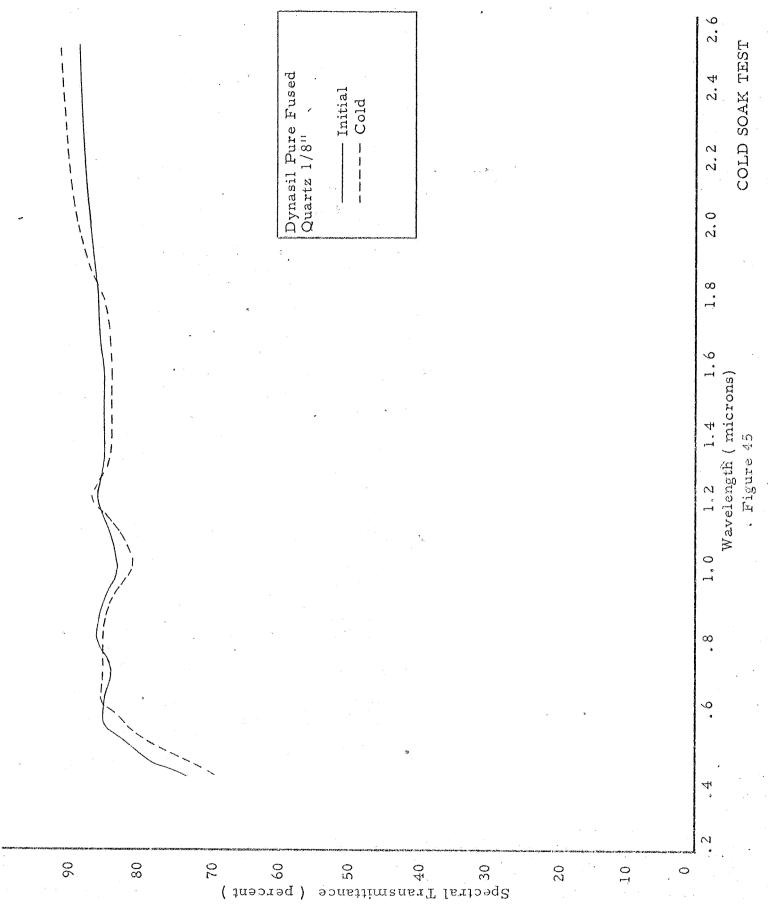
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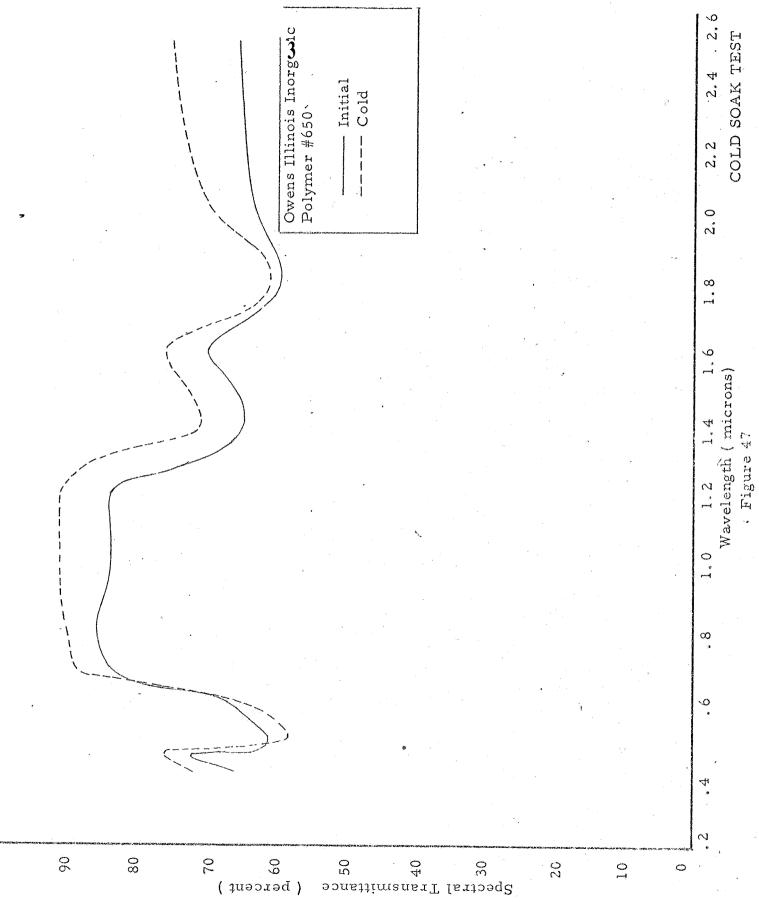
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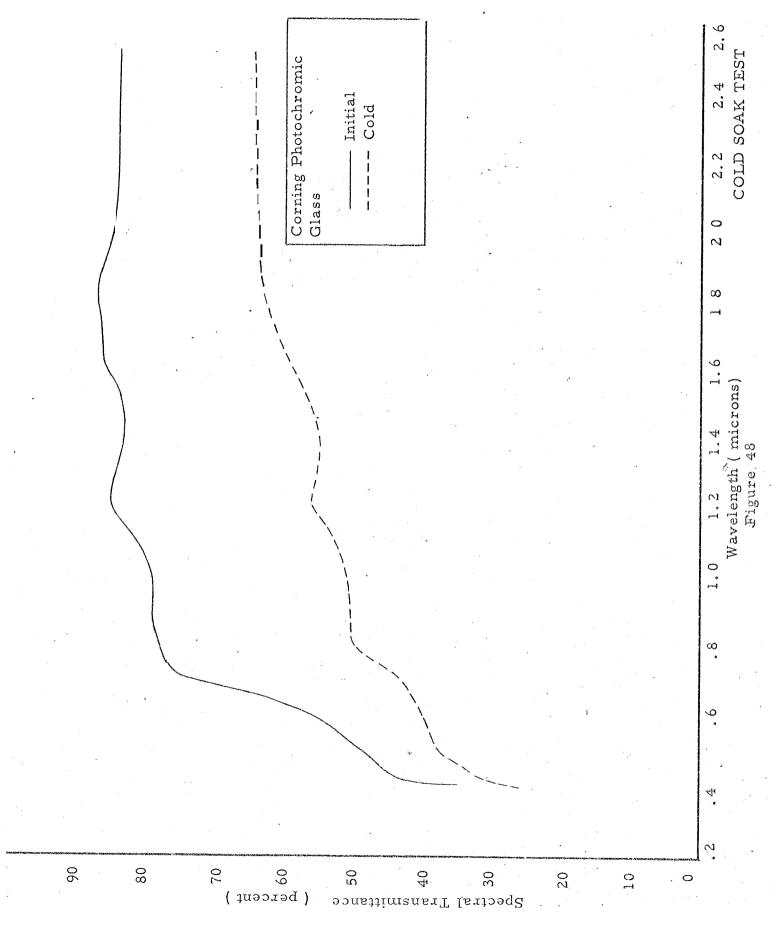


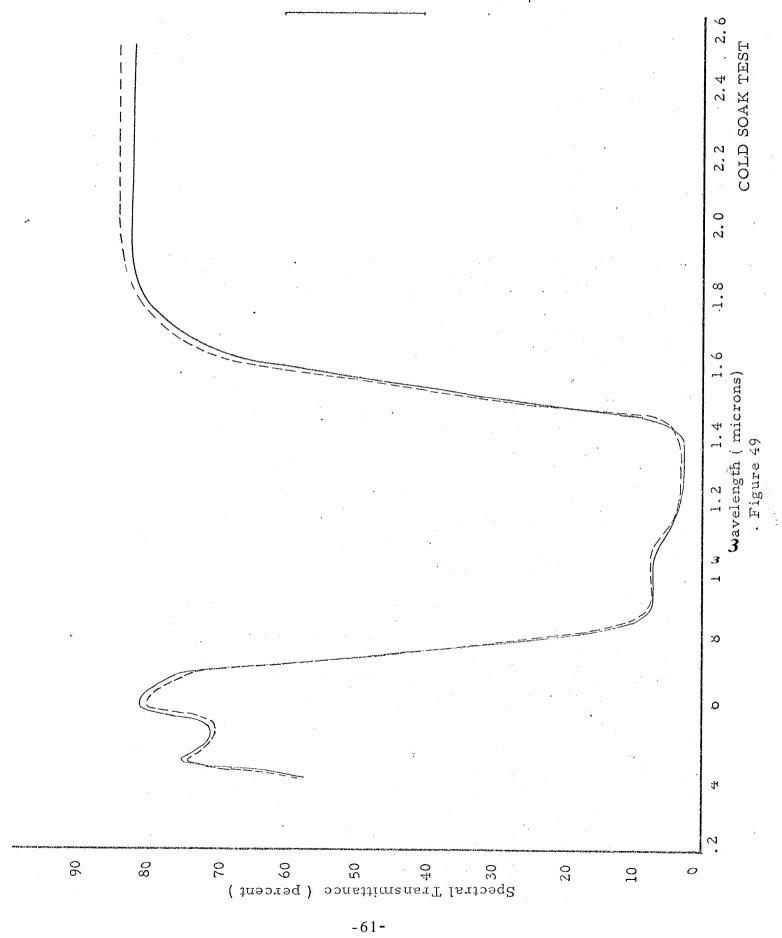


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VI. CONCLUSIONS

The test showed that the glasses were stable with temperature and solar radiation while the plastics and polymers would not withstand the temperature excursions and either deformed and/or darkened to such an extent that the transmissibility went to zero. Each individual material will be discussed in the following pages. Photographs of the actual materials used in the first teat are shown in Figure 50 and those of the second test are shown in Figure 51.

Polymer 650

In both tests this material appeared unstable under temperature cycling and ultraviolet irradiation. The samples cracked in both tests and it appears the transmissibility increased from the original transmission. Apparently there was a lens effect due to the distortion of the sample which focused the light on the collecting lens on the exit port of the chamber and increased the light to the monochromator.

Polymer 100

This material also appeared unstable due to the temperature excursions and ultraviolet exposure. In the first test the sample cracked and the visible and infrared cutoff property of the sample was destroyed. The second sample did not crack, however, it did degrade considerably and finally distorted so that its transmissibility appeared to also increase. An immediate check of the stability of the detector was performed to insure that there was no change due to the detector shifting. The detector was stable within the limits of the stated accuracy of the equipment.

Pure Fused Quartz 1/8" and 1/4"

There was no degradation of the quartz samples of both thicknesses. The second test confirmed that there was no appreciable degradation to the samples. It may be seen that there was a film or discoloration to the first test sample which is not present on the

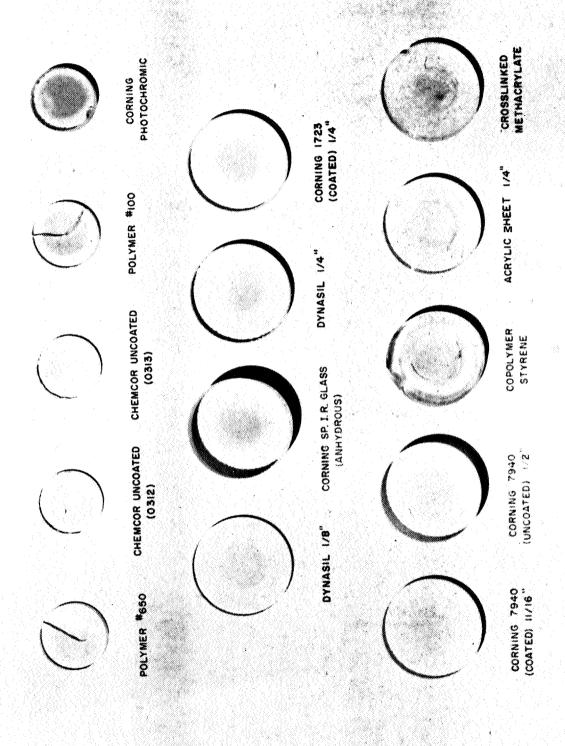


Figure 50 -- First Test Samples - After Exposure

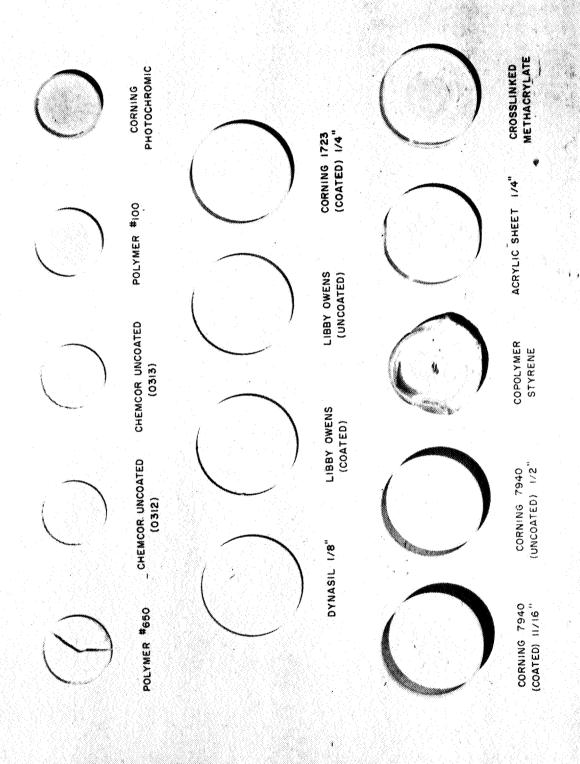


Figure 51 -- Second Test Samples - After Exposure



second sample. The 1/4" quartz sample also showed some decrease in transmittance but it is also believed this is due to the film which shows on the surface of the sample.

Corning Photochromic

This glass has the property that its spectral transmittance is a function of the spectrum and intensity of the incident light so that the curves which were obtained were very erratic in both tests. In the cold soak the photochromic showed the most degradation as a result.

Corning 7940 (Coated)

In the first test there was a decrease in the transmissibility in the visible and infrared portions of the spectrum where the transmission is highest probably due to a discoloration on the glass which may be seen. In the second test there was very little difference between the initial and final transmittance of the glass in both of these regions with a conclusion that there was no degradation of this glass due to the irradiation or temperature. The variations in the 14, 45, and 60 day transmission of higher transmission are considered to be within the system accuracy. There was no obvious mechanical deformation of the glass as was observed in some of the other materials.

LEM Chemcor Uncoated Code 0312

The conclusion for this material is that it did not degrade as a result of this test. Both the first and second test showed the transmission to be well within the accuracy band of the equipment. Examination of the samples after removal from the chamber showed no mechanical deformation or visible evidence of damage. A slight film existed on the sample in the first test but was not present on the sample in the second test

Acrylic Sheet 1/4"

This material did not withstand the temperature excursions and electromagnetic irradiation. Degradation to almost a completely



opaque material occurred after the 45 days and before 60 days in the first test and after the initial measurements in the second test.

New Chemcor Uncoated Code 0313

There was no discernible degradation of this material due to this test.

Co-Polymer Stryene

The first sample in the first had high transmission cutoff in the visible region and a cutoff in the infrared region. This material successively degraded until it became completely opaque at the conclusion of the test. The second sample in the second test also degraded to practically zero transmission; however, it did not exhibit this sharp cutoff in the infrared region initially as can be seen from the curve of Figure 26. In the photographs of the samples it may be seen that this material exhibited considerable mechanical deformation in both tests with charring of the exposed surfaces in both tests.

Cross-Linked Methacrylate 1/411

In both 90-day tests this material completely degraded to almost complete opacity and exhibited mechanical deformation. In the first test, the complete degradation occurred after the 14th day transmission measurement; in the second test the transmission went from 90% to 10% after the initial measurement and before the 14th day measurement. By the 45th day the material was completely opaque. Successive measurements showed almost zero transmission in both cases.

Corning 7940 (Uncoated) 1/2¹¹

It must be concluded there was no degradation of this material in both tests since the initial and final transmissions were very nearly the same.



Corning 1723 (Coated) Inner Pane 1/411

There was no degradation of this material. Again in the first test there was a film deposited on the glass which probably accounted for the variations in transmission during the test and a wider spread of measurements than during the second test. The second test showed no degradation and actually a slight increase in transmission which could be due to either the glass being cleaner at the end of the test than at the beginning due to the initial outgassing of the plastics or just within the accuracy of the system.

Libby Owens (Coated)

It must be concluded that there was no degradation of this material due to the test. This was run only in the second test. Although the data showed a 5 - 6% seduction in transmittance this would be within the accuracy of the system, There was no visible discoloration or mechanical deformation of the glass sample.

Corning Special IR Glass (Anhydrous Composition)

There was a slight absorption band developed at 1.0 micron which showed up in three measurements - the 60-day, the cold soak and the 90-day final measurement, There was no appreciable degradation besides this at the other wavelengths.

Libby Owens (Uncoated)

There was no degradation of this material as a result of the test. It was only run in one test, the second test. Visible examination of this material did not reveal any discoloration or mechanical deformation.



Cold Soak

Since the only glass which showed appreciable degradation was the photochromic and there was no way to control the amount or spectrum of light incident on the sample from other sources, it was concluded that the extreme cold temperature had no effect on the transmittance of the materials. The other materials showed either no difference or very little difference within the error band of the system.



VII. REFERENCES

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